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Field Screening Methods Catalog: User's Guide

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The Field Screening Methods Catalog (FSMC) is a technical document produced by the United States Environmental Protection Agency's Office of Emergency and Remedial Response — Analytical Operations Branch. This document is a compilation of methods that were identified as being used in EPA Regions. Several methods contain no method performance information, because this information was not available. The methods are provided as submitted by the technical contact listed in the method description. The Analytical Operations Branch has not evaluated the methods contained in this document, but has simply compiled existing methods.

The Catalog was developed to assist the user in identifying field screening methods applicable to specific site characteristics. The computer system was developed using dBase III Plus and operates on an IBM compatible microcomputer. The user may search for field methods by entering selection criteria, including chemical class, name, or CAS number, method name or number, matrix type (air, soil, and/or water) and a minimum detection limit.

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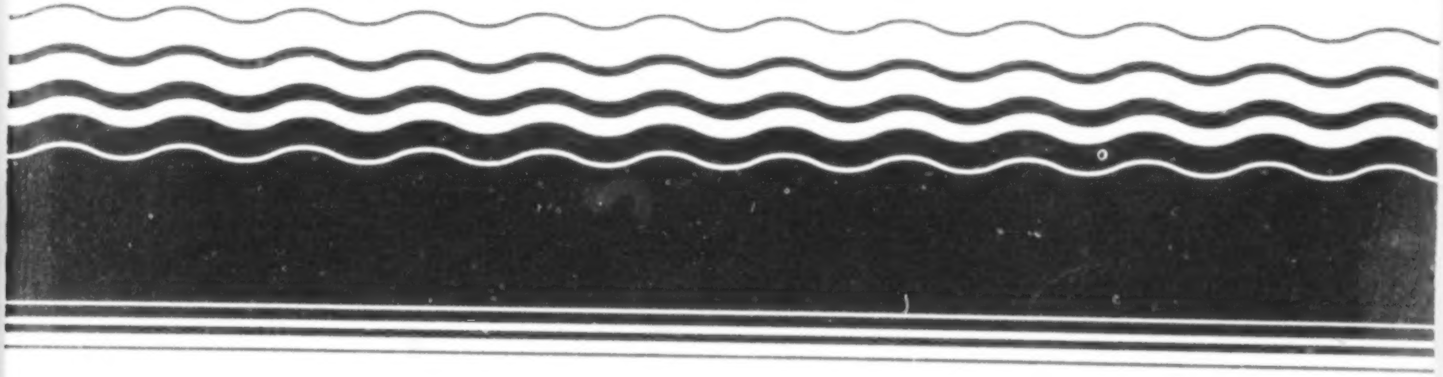

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Office of Emergency and Remedial Response
Hazardous Site Evaluation Division
U.S. Environmental Protection Agency
Washington, DC 20460

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EXECUTIVE SUMMARY

The Field Screening Methods Catalog (FSMC) is a technical document produced by the United States Environmental Protection Agency's Office of Emergency and Remedial Response — Analytical Operations Branch. This document is a compilation of methods that were identified as being used in EPA Regions. Several methods contain no method performance information, because this information was not available. The methods are provided as submitted by the technical contact listed in the method description. The Analytical Operations Branch has not evaluated the methods contained in this document, but has simply compiled existing methods.

The methods presented in this Catalog should not be viewed as Standard Operating Procedures (SOP) but rather as a compilation of *available technologies* which have been successfully utilized on a site-specific basis. Prior to the application of any of these methods, the user is urged to consult with the technical contact listed for each method, and an analytical chemist familiar with both the instrumentation/method and the specific conditions inherent to the site under consideration. It is also critical that an assessment be made of these site specific conditions, and how they may effect the utility of the method and the resulting data quality.

The FSMC is available to users, particularly those individuals responsible for developing and overseeing sampling activities at Superfund hazardous waste sites, e.g., Regional Project Managers (RPMs) and contractor Site Managers (SMs). The Catalog currently consists of a User's Manual including a listing of all methods, a "pocket guide," a field screening methods data base and a computerized information retrieval system contained on two floppy discs. The discs are not included with this User's Guide but will be distributed separately to EPA Regional Offices for further distribution as the Region sees fit. Information on how to acquire copies of the discs is available from the EPA Headquarters FSMC Systems Coordinator — Analytical Operations Branch (WH548A).

The Catalog was developed to assist the user in identifying field screening methods applicable to specific site characteristics. The computer system was developed using dBase III Plus and operates on an IBM compatible microcomputer. The user may search for field methods by entering selection criteria, including chemical class, name, or CAS number, method name or number, matrix type (air, soil, and/or water) and a minimum detection limit.

It is important to note that the methods presented in this Catalog are an option available to RPMs and SMs; the methods are not designed to "take the place" of other analytical options. They are intended to supplement existing methods and provide options based on the specific needs of the sampling/analytical activity.

Based on the anticipated momentum that the FSMC will generate within the Agency and in the private sector, this Catalog is provisional and is expected to be revised and updated as more information becomes available on existing screening/analytical techniques, and as new techniques are developed, tested and applied to field analysis at hazardous waste sites.

In order to develop a historic data base on the range of data quality achieved by each method it is critical that users conduct sufficient QC analysis to allow a determination of data quality as defined by precision and accuracy. Additional information which will assist in defining the overall utility of the method includes method detection limits (sensitivity determination), matrix effects, interferences, as well as any sample preparation/processing specifications and overall operator assessment of the method. It is requested that as this type of information is developed, it be forwarded to the FSMC System Coordinator. A User Comment form has been included in Appendix C.

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1.0 INTRODUCTION AND BACKGROUND

The United States Environmental Protection Agency (EPA) awarded the first two major hazardous waste contracts in 1979. Since that time, the scope and nature of hazardous waste investigations have grown and matured. The culmination of this maturation process is the present day Superfund Program, as amended by the Superfund Amendments and Reauthorization Act of 1986 (SARA).

Throughout this period of maturation, the basic initial (yet complex) premise of field investigations has remained the same — to determine the chemical constituents (identity and quantitation) in environmental media (air, soil, water) as well as "source" media such as sludges, and materials from containers including drums and tanks. An additional complexity, which became evident as field investigations were initiated, was the frequent need for "rapid turnaround" of sample analysis results. This was critical, for example, to evaluate if emergency response was necessary.

The Contract Laboratory Program (CLP) was established by EPA to provide analytical support for the massive amount of data that was being generated by the numerous hazardous waste sites throughout the country. This program provides a range of analytical chemistry services of known quality on a high-volume, cost-effective basis. The central and overriding assumption governing the structure and function of the CLP is the basic requirement to provide legally-defensible analytical results for use in supporting Agency actions. As of early 1987, the CLP was "able to provide over 6,000 sample analyses per month through its Routine and Specialized Analytical Services (RAS and SAS) Programs". Based on the "central and overriding assumption....to provide legally-defensible analytical results..," CLP data deliverable packages are accompanied by very specific documentation containing information which includes initial and continuing calibration, GC/MS tuning, surrogate percent recovery, matrix spike duplicate results, GC chromatograms, Furnace AA analysis, digestion/distillation logs, ICP interference and serial dilution analysis, and spectra for every sample and every blank, standard, or spike run with a particular set of samples. Data delivery for this complete data package takes approximately 35-40 days after submission. An additional 30 days for Regional review of each data package is also required. Faster turn-around times can be achieved through the SAS and through expedited review by the Regions.

It is now recognized in the scientific/regulatory community that, frequently, the approximately 65-70 day turn-around in sample analyses is unacceptable, and that the "quality" of data provided by the CLP is not required for all samples. Two distinct developments within EPA address this concern: first, the increasing use of field screening/analytical methods, and second, the issuance of the *Data Quality Objectives for Remedial Response Activities* (EPA, 1987 - OSWER Directive 9355.0-7A). The development of field analytical methods was initiated early in the hazardous waste program history. The FITs developed and used field analytical techniques, first for health and safety related (air) monitoring and subsequently for sample screening and analyses of increasing complexity. This use of field methods was frequently performed in conjunction with EPA laboratories and the Environmental Response Teams (ERTs). As such, initial use of instruments such as the Foxboro Organic Vapor Analyzer (OVA) and the HNu Systems HNu 101 were limited to air monitoring to determine levels of safety protection. This initial use of field instrumentation made the FITs and ERTs familiar with the capability of these and other instruments and paved the way for their use as analytical tools in the Superfund Program.

The second development, while discussed for many years, was initiated and codified in 1986 with the issuance of a number of EPA sponsored documents, the foremost of which were the *Data Quality Objectives for Remedial Response Activities* (EPA, 1987 - OSWER Directive 9355.0-7A) and *A Compendium of Superfund Field Operations Methods* (EPA 1987a - OSWER Directive 9355.0-14). Data Quality Objectives (DQOs) recognize and promote the concept that in the course of a typical Superfund remedial investigation, samples are taken with various objectives and that these various objectives require different "data quality". In other words, not all objectives require, for example, CLP quality data. While DQOs present a framework for identifying and achieving site specific data quality objectives through the appropriate sampling and analytical techniques, the Compendium "focuses primarily on (sampling) techniques and methods used during the fieldwork phase of a remedial investigation."

DQOs define five levels of analytical support which are available for sample analysis. These levels are based on numerous factors, including and foremost, data quality requirements. These five levels of analytical support are defined as Levels I through V and range from hand-held equipment and screening techniques to sophisticated GC/MS instrumentation and analysis. Specific discussions of these analytical levels, their applicability, and limitations, as well as specific directions for use are contained in both the *Compendium of Superfund Field Operations Methods* and in the *Data Quality Objectives for Remedial Response Activities* (EPA, 1987a - OSWER Directive 9355.0-14, 1987 - OSWER Directive 9355.0-7A). In addition, a brief overview of these five analytical levels is included in Sections 1.4 and 1.5. It should be noted that no clear delineation exist, especially between Levels I and II analysis. As a general rule-of-thumb, Level I analysis typically result in qualitative measurements for the presence or absence of classes of contaminants (typically volatile organics, although specific compounds can also be measured in certain instances), while Level II analyses can provide qualitative and frequently quantitative values for both groups and specific analytes. At times, the same instrument can be used for both Levels I and II analyses. For example, an OVA (a flame ionization organic vapor detector) can be used in the "survey mode" for Level I analyses to measure total organic vapor. In the GC mode, this same instrument can be used to obtain Level II data of specific analytes.

1.1 FIELD SCREENING METHODS CATALOG

One of the primary reasons for Superfund (as well as RCRA and other EPA programs) contamination monitoring at potential and known hazardous waste sites is the determination of the identity, concentrations and vertical and horizontal extent of chemical contamination. An essential element of any monitoring effort is analytical support that provides rapid sample throughput while matching the data quality objectives of the sampling effort.

Experience gained during the first five years of the Superfund program has shown that various types of field screening/analytical methods are suitable and are being used during field investigations to characterize "hot spots," evaluate the necessity of emergency response, define general site conditions, assist in well placement and screen setting, aid in the selection of sampling locations, compare off- and on-site conditions, estimate potential population exposures, determine the completeness of cleanup actions (such as excavations), and establish long-term monitoring.

Presently, field screening/analytical techniques are routinely being used throughout the Superfund program. However, because of the decentralized nature of the program, knowledge and skills gained in one region or state, or even at one site, are not necessarily transferred to others. Many personnel responsible for developing/overseeing site sampling efforts need to have timely and accurate information regarding the availability of appropriate analytical methods. To meet this need and to facilitate transfer of information about methods for measuring and screening chemicals in the field, the United States Environmental Protection Agency has developed the Field Screening Methods Catalog (FSMC).

To date, thirty-one methods have been compiled and documented in the Catalog. Of the thirty-one methods contained in this Catalog, four are in the developmental stage (Use of Bonded Sorbents for Pesticide Analysis, Use of Bonded Sorbents for Semi-Volatile Analysis, Immunoassays for Trace Organic Analysis, and Use of Fiber Optic Sensors in Environmental Monitoring) and one (Trace Atmospheric Gas Analyzer - TAGA) cannot truly be considered a viable "field method" due to its size, limited availability, and cost. However, since these methods represent an available technology they have been retained in this Catalog in a separate section. These five methods have not been segregated in the computerized data base and as such will be selected when the appropriate parameters are input. In considering the applicability of these five methods, the user should be aware of the above mentioned limitations, and take sufficient QA/QC steps to allow an assessment of data quality to be performed.

For the purposes of this Catalog, the term "field methods" is used as a catch-all phrase and includes methods which utilize hand held instrument and/or instruments which can be carried with relative ease, portable instruments which can be set up and used in the back of a van or field trailer, and fieldable instruments which usually require a more stationary and stable environment such as a field (mobile) laboratory. These terms are defined in more detail in Section 1.5.

The methods presented in this Catalog include analyses for metals, volatile and semi-volatile organics, phenols, pesticides, PCBs, dioxins and polycyclic aromatic hydrocarbons. In addition, several soil gas sampling techniques have been included because of the expanding use of such techniques.

The FSMC consists of a reduced "pocket guide," a computerized retrieval system stored on two floppy disks and a user's guide. The pocket guide was designed for field personnel and provides a concise description of each method which provides field staff with the information needed to consider the range of analytical methods that might be appropriate for the site. The computer program is written on dBase III, is IBM compatible, and provides search capabilities according to chemical of interest (class, name or CAS number) or method name or number. The search options also prompt the user to select a matrix of interest (air, soil and/or water) and a detection limit to match and provide an available method that will meet the need of the user as closely as possible. Once a method or methods that can potentially meet the user's need is found, a number of options are available to view and/or print the method(s). These options are discussed in more detail in Section 4.0. In addition, this volume provides an introduction, a computer user's guide, including an appendix containing copies of the thirty-one methods.

For ease of use and consistency, a standard set of "Fields" (or major headings) have been developed to organize method specific information. These information provided for each method include the following:

- Method name and number (number is specific to this Catalog);
- Summary and method description;
- Application, limitations, and instrumentation used;
- Performance specifications such as detection limits, selectivity, accuracy, precision and repeatability;
- Use of the method (location, CERCLIS site number [where appropriate], and matrix);
- Preparation, maintenance, and cleanup;
- Calibration;
- Analysis time;
- Capital costs; and
- Source of technical information.

It is important to note that these fields specify the parameters that have been utilized in the development and subsequent use of the methods and not necessarily the only possible choices. For example, the Instrument Used field for PCB Analysis Using a Gas Chromatograph in an On-Site Laboratory - Hexane Extraction, shows that a Hewlett-Packard 5880 Gas Chromatograph with an electron capture detector was used. However any GC with analogous capabilities could be used to perform this analysis. Caution should however be used to assure that modifications to the method will not effect the quality of the resulting data. As such it is important to consult with an analytical chemist prior to modifying any of the methods contained in this Catalog.

1.2 INTENDED USERS

The FSMC system has been developed for individuals who are responsible for developing or overseeing sampling activities at Superfund hazardous waste sites. As already mentioned, these individuals require information regarding analytical/screening techniques which have demonstrated field utility and which complement those methods used by the CLP.

The FSMC addresses the need of site personnel to determine what field methods, if any, are most appropriate in a given situation. Based on preliminary site information such as types of contaminants, the user can identify field analytical/screening methods that have been developed and applied at other similar sites and therefore may be of use for site characterization and other screening/analytical requirements.

1.3 SYSTEM UPDATES

Documentation and system revisions will be made to the pocket-guide, user documentation, and computer retrieval system. The documentation updates will focus on the user documentation and the method descriptions to ensure accuracy and completeness of available methods, and additions of new methods on a periodic basis.

1.4 TECHNICAL CONSIDERATIONS

The appropriate type of sampling and analysis at a given site depends on numerous factors, the foremost of which are the intended end use of the data and associated data quality requirements. Data quality, as stated in the *Data Quality Objectives for Remedial Response Activities* (EPA, 1987 - OSWER Directive 9355.0-7A), is defined by the level of analytical support appropriate to various data uses. As such, five levels of analytical support (Levels I-V) are defined below and are appropriate to a number of overlapping data uses as shown on Tables 2-1 and 2-2.

- *LEVEL I* - Field Screening or analyses using portable instruments. Results are often not compound specific and not quantitative but results are available in real-time. It is typically the least costly of the analytical options.
- *LEVEL II* - Field analyses using more sophisticated field portable analytical instruments. In some cases, the instruments can be set up in a mobile laboratory on site. There is a wide range in the quality of data that can be generated from qualitative to quantitative.
- *LEVEL III* - Laboratory analysis using methods other than the CLP-RAS. This level is used primarily in support of engineering studies using standard EPA approved procedures. Some procedures may be equivalent to CLP-RAS, but without the CLP requirements for documentation.
- *LEVEL IV* - CLP Routine Analytical Services (RAS). This level is characterized by rigorous QA/QC protocols and documentation and provides qualitative and quantitative analytical data. (Some Regions have obtained similar support via their own regional laboratories, university laboratories, or other commercial laboratories).
- *LEVEL V* - Non-standard methods. Analyses which may require method modification and/or development. CLP Special Analytical Services (SAS) are considered Level V.

Whereas the "quality" of data generally increases from Level I through Level V as shown above, this can only be stated in a general way and in certain instances and with the appropriate QA/QC procedures, the "quality" of Level II and III data can parallel that achieved by Level IV and V data. Consider for example a Level II analysis for volatile organics using a GC in a field laboratory; in this situation, the "quality" of the data may be higher than that achieved in an offsite laboratory (Level III, IV, or V) due to sample handling considerations. Similarly, Level II and III analyses can at times provide lower detection limits than those required by Level IV requirements, and as such, the "quality" of data is higher based on the data quality objectives.

It is important to note that in most situations the exact "quality" of an analysis cannot be specified or determined prior to the analysis and as such sufficient QA/QC steps have to be taken (e.g., documentation of blank injections, calibration standard runs, runs of qualitative standards between samples, and analysis of duplicates and spikes) to be able to assess the quality achieved. As such, one of the major benefits of Level IV analysis is that it provides sufficient documentation to allow (qualified) personnel to review and evaluate data quality. In other words, while the "quality" of Level IV data may not be higher (as defined by the precision and accuracy) than those achieved by other levels of analysis, it does provide data of "known quality"

TABLE 2-1
SUMMARY OF ANALYTICAL LEVELS APPROPRIATE TO DATA USES

DATA USES	ANALYTICAL LEVEL	TYPE OF ANALYSIS	LIMITATIONS	DATA QUALITY
SITE CHARACTERIZATION MONITORING DURING IMPLEMENTATION	LEVEL I	TOTAL ORGANIC/INORGANIC VAPOR DETECTION USING PORTABLE INSTRUMENTS	· INSTRUMENTS RESPOND TO NATURALLY-OCCURING COMPOUNDS	· IF INSTRUMENTS CALIBRATED AND DATA INTERPRETED CORRECTLY, CAN PROVIDE INDICATION OF CONTAMINATION
SITE CHARATERIZATION EVALUATION OF ALTERNATIVES ENGINEERING DESIGN MONITORING DURING IMPLEMENTATION	LEVEL II	· VARIETY OF ORGANICS BY GC; INORGANICS BY AA; XRF · TENTATIVE ID; ANALYTE-SPECIFIC · DETECTION LIMITS VARY FROM LOW ppm TO LOW ppb	· TENTATIVE ID · TECHNIQUES/INSTRUMENTS LIMITED MOSTLY TO VOLATILES, METALS	· DEPENDENT ON QA/QC STEPS EMPLOYED · DATA TYPICALLY REPORTED IN CONCENTRATION RANGES
RISK ASSESSMENT PRP DETERMINATION SITE CHARACTERIZATION EVALUATION OF ALTERNATIVES ENGINEERING DESIGN MONITORING DURING IMPLEMENTATION	LEVEL III	· ORGANICS/INORGANICS USING EPA PROCEDURES OTHER THAN CLP CAN BE ANALYTE-SPECIFIC · RCRA CHARACTERISTIC TESTS	· TENTATIVE ID IN SOME CASES · CAN PROVIDE DATA OF SAME QUALITY AS LEVELS IV, NS	· SIMILAR DETECTION LIMITS TO CLP · LESS RIGOROUS QA/QC
RISK ASSESSMENT PRP DETERMINATION EVALUATION OF ALTERNATIVES ENGINEERING DESIGN	LEVEL IV	· HSL ORGANICS/INORGANICS BY GC/MS; AA; ICP · LOW ppb DETECTION LIMIT	· TENTATIVE IDENTIFICATON OF NON-HSL PARAMETERS · SOME TIME MAY BE REQUIRED FOR VALIDATION OF PACKAGES	· GOAL IS DATA OF KNOWN QUALITY · RIGOROUS QA/QC
RISK ASSESSMENT PRP DETERMINATION	LEVEL V	· NON-CONVENTIAL PARAMETERS · METHOD-SPECIFIC DETECTION LIMITS · MODIFICATION OF EXISTING METHODS · APPENDIX 8 PARAMETERS	· MAY REQUIRE METHOD DEVELOPMENT/MODIFICATION · MECHANISM TO OBTAIN SERVICES REQUIRES SPECIAL LEAD TIME	· METHOD-SPECIFIC

TABLE 2-2
APPROPRIATE ANALYTICAL LEVELS - BY DATA USE

DATA USE / ANALYTICAL LEVEL	SITE CHARACTERIZATION (INCLUDING HEALTH & SAFETY)	RISK ASSESSMENT	EVALUATION OF ALTERNATIVES	ENGINEERING DESIGN OF REMEDIAL ACTION	MONITORING DURING IMPLEMENTATION OF REMEDIAL ACTION	PRP DETERMINATION	OTHER ____
LEVEL I	✓				✓		
LEVEL II	✓		✓	✓	✓		
LEVEL III	✓	✓	✓	✓	✓	✓	
LEVEL IV		✓	✓	✓		✓	
LEVEL V		✓				✓	
OTHER							

NOTE: CHECK APPROPRIATE BOX (ES)

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The decision to use field analytical/screening techniques (i.e., Levels I and II) must be made on a site specific, and on a sampling-event-by-sampling event basis. Factors to be considered include data quality requirements, parameters for which the analytical method is valid, contaminants of concern, required detection limits, and the range of precision, accuracy, representativeness, completeness, and comparability (PARCC) required and provided by each analytical option. Documentation and chain-of-custody considerations are also relevant and should be considered.

While representativeness, completeness and comparability are, for all practical purposes, sampling considerations, precision and accuracy encompass both sampling and analytical considerations and define the quality of the resulting data. Precision measures the reproducibility of measurements under a given set of conditions. Specifically, it is a quantitative measure of the variability of a group of measurements compared to their average value. Precision is usually stated in terms of standard deviation but other estimates such as coefficient of variation (relative standard deviation), range (maximum value minus minimum value), and relative range are also common. Since precision defines the scatter of results about a mean value, a lower standard deviation means less scatter.

The overall precision of measurement data is a mixture of sampling and analytical factors. Analysis of field and laboratory replicates provides a measure of overall precision.

Accuracy measures the bias in a measurement system. Sources of error are the sampling process, field contamination, preservation, handling, sample matrix, sample preparation and analysis techniques. Sampling accuracy can be assessed by measuring the concentration of contaminants in field/trip blanks, while analytical accuracy may be assessed through use of known and unknown QC samples and matrix spikes.

Accuracy is most frequently reported as percent recovery, or percent bias. A 100% recovery indicates a completely accurate measurement; the greater the deviation (i.e., under or over) is from 100%, the less accurate the measurement. Percent bias reports the difference of the result from the true value. A completely accurate measurement would have zero percent bias; the lower the percent bias, the more accurate the measurement.

While historical accuracy and precision information which is classified by media are available for CLP analytical methods (Level IV), significantly less information exists for the other levels of analysis (including Level V). Historic accuracy and precision information has been compiled and classified by media and by analytical level (EPA, 1987 - OSWER Directive 9355.0-7A). This information is excerpted and presented in Appendix B.

The available data base for documenting accuracy and precision (as well as minimum detection limits) for Levels I and II is sparse. To supplement the sparse and scattered information which exist in published and unpublished reports etc., personnel who use the methods presented in the FSMC are encouraged to catalog the precision and accuracy that is obtained during his/her specific use, and to provide this information to the FSMC Systems Coordinator.

Based on the required data quality objectives and data uses, the choice to use field screening/analytical methods should be made in conjunction with a "confirmation" program to include a rigorous QA/QC program (analysis of QC samples), instrument calibration, and Level III or IV confirmation. The user is urged to consult both the *Data Quality Objectives for Remedial Response Activities* and the *Compendium of Superfund Field Operations Methods Manuals* (EPA, 1987 - OSWER Directive 9355.0-7A and EPA, 1987a - OSWER Directive 9355.0-14) as part of the decision process used to define the appropriate levels of analytical support. It is further recommended that, based on the intended data uses and associated data quality requirements, the data obtained through field analysis/screening be confirmed by CLP analysis.

1.5 OVERVIEW OF LEVELS I AND II ANALYSES

Level I and II analyses are defined as field screening/analytical methods which utilize equipment amenable to the rigors of field conditions, and are located at or near the sampling site.

Level I analytical support is typically defined as field screening, with the objective of generating data which will generally be used (for example during Phase 1 investigations), in refining sampling plans and determining the extent of contamination. A second objective of Level I analyses is to conserve other analytical support resources.

Level I analyses are generally effective for total vapor readings using portable photoionization or flame ionization detectors (PID or FID) which respond to a variety of organic and inorganic volatile compounds. Detection is typically limited to volatile compounds. These types of analyses provide data for on site, real time total vapor measurements, evaluation of existing conditions, sample location optimization, extent of contamination, and health and safety evaluations. Data generated from Level I analyses are considered qualitative in nature although semi-quantitative and/or quantitative data can be generated, for example, by using the GC option of a FID, with sufficient calibration. Data generated from Level I analyses provide the following:

- Identification of soil, water, air and waste locations which have a high likelihood of showing contamination through subsequent analysis;
- Real-time data to be used for health and safety considerations during site investigations;
- Qualitative data relative to a primary calibration standard if the contaminants being measured are unknown;
- Quantitative data if a contaminant is known and the instrument is calibrated to that substance; and
- Presence or absence of contamination.

Level II analytical support is designed to provide real-time data for ongoing field activities or when initial data will provide the basis for seeking laboratory analytical support. As such, Level II analytical methods can be effectively utilized when a phased approach is used for field sampling. There have also been a significant number of instances where data derived from Level II support have been used to make decisions about site disposition.

Field analysis using Level II analytical options can provide data from the analysis of air, water, soil, and waste materials for many Target Compound List (TCL) organic compounds including volatiles, base neutral acid (BNA), extractable organics, and pesticides/PCBs. Inorganic analysis can also be conducted using field atomic adsorption (AA) and other instruments.

Level II analyses are used for on site, real-time baseline data development, extent of contamination and remedial activities and generally provide rapidly available data for a variety of activities including hydrological investigations (establish depth/concentration profiles); extent of contaminant determination including special activities such as vadose zone sampling; cleanup operations (determine extent of contaminated soil excavation), and health and safety considerations.

Typically, a gas chromatograph and more sophisticated instruments operated in the field provide the bulk of the analytical support at this level. The ability to assess the data quality (accuracy and precision) is dependent upon the QA/QC steps taken in the process, including documentation of blank injections, calibration standard runs and runs of standards between samples.

The level of precision and accuracy that can be achieved by a specific Level I or II analysis varies as a function of numerous factors including the matrix and contaminant being sampled, and perhaps most importantly, the skill of the analyst doing the work. As summarized in the *Data Quality Objectives for Remedial Response Activities* (EPA, 1987 - OSWER Directive 9355.0-7A) "because the procedures (for Level II) are not formalized, a great deal of improvisation usually takes place. The inherent variability of the procedures themselves would make the development of a centralized quality assurance data base tenuous. The same reasoning would apply to making uncertainty predictions based on a centralized data base". On the other hand, Level II analyses based in a mobile laboratory setting and using state of the art sophisticated instrumentation, can allow mobile laboratory capabilities to approach the analytical range and performance (accuracy, sensitivity and precision) achieved in off-site analytical facilities.

Based on the undocumented data quality of most Level I and II analyses, data generated in the field are typically confirmed by submitting duplicate samples to the CLP. Although no statistical methods are available to determine the exact number of samples to submit for confirmation, numerous factors have to be considered including:

- Objective of sampling (i.e., data quality objectives);
- Data uses, and
- Method of analysis used and the level of accuracy and precision achieved.

In general, confirmation samples should include a subset (or all) of designated critical samples, a subset of samples covering the entire range of identified concentrations, and a subset of samples near the action level and near the "0" concentration or not detectable range.

An additional factor to consider is the measured precision of the field instrument in use. When high precision is measured, less samples need to be confirmed; if however, a low precision is calculated, it is recommended that based on the data quality objectives defined for the site, the analysis be suspended until a qualified chemist determines the reason for the low precision.

The equipment utilized for Level I and II analyses can vary greatly in terms of physical configuration, size, weight, power requirements, and the level of accuracy and precision achievable. Based on the mobility of the equipment, three terms are typically used to describe the mobility of the equipment:

> **Portable:** Requires no external power requirements; hand held devices which can be easily carried by one person. Typically includes photoionization detection (PID) and flame ionization detection (FID) to measure the total amount of ionizable materials, mostly volatile organic compounds. Also includes suitcase-sized gas chromatographs (GC) with assorted detectors and capabilities, and X-Ray fluorescence devices.

Fieldable: Particularly rugged; limited external power required. Easily transported in a van, pick-up, or four wheel drive.

Mobile: Small enough to carry in a mobile lab; includes most analytical instruments. Power considerations may limit the use of many instruments in mobile laboratories.

As outlined in this brief discussion of Level I and II analytical methods, numerous factors must be considered when determining which method should be implemented at a site. Data quality objective for the sampling event in question and the ultimate end use of the data developed should also be considered.

2.0 INSTALLING THE SYSTEM

The first time the FSMC system is run, an installation menu will appear and request information about your computer. This system will run on a computer with two floppy disk drives or one floppy disk drive and at least one hard drive. The floppy disk drive on the hard disk system is used to copy all programs and data files to the hard drive (usually C:), it is not used to run the system once all programs and data files have been copied to the hard drive.

It is assumed that the computer used has already been turned on and loaded with MS-DOS version 2.0 or higher. Consult your computer's operating manual for these procedures.

Some users will have to change the CONFIG.SYS file to properly run the compiled dBASE III code. The CONFIG.SYS file should have the following two lines:

```
BUFFERS=25
FILES=20
```

To list the file, enter

```
C> TYPE \CONFIG.SYS
```

If the two values are equal to or larger than listed above, then no changes are needed. If the values are less than those listed above, or if the lines are missing, the file will have to be edited. Please consult your MS-DOS manual for instructions on editing this file.

2.1 INSTALLATION ON A TWO DISK DRIVE COMPUTER SYSTEM

Users with a dual-floppy disk drive system may use FSMC. The following procedures should be followed the first time the system is used so that the FSMC will recognize which disk drives will be used.

The user should make a copy of the two system diskettes and return the originals to the manual for any future installations.

1. Insert FSMC disk A into drive A and
   disk B into drive B.
2. If the A: drive is not the current drive, enter
   B:>A: [RETURN/ENTER]

3. To start the FSMC system, enter
   ```
   A:>FSMC        [RETURN/ENTER]
   ```
4. When asked "Do you have a hard drive [Y/N] ?," enter
   ```
   N              [RETURN/ENTER]
   ```

The installation procedure will take about one minute. After the installation is completed, the main menu will appear.

2.2 INSTALLATION ON A HARD DRIVE COMPUTER SYSTEM

Throughout this manual it is assumed that the hard disk is named "C:" and the directory on which FSMC resides is "C:\FSMC". Other combinations are valid and should be designated during the installation process.

1. Create a directory on your hard disk
   ```
   C:>MKDIR \FSMC        [RETURN/ENTER]
   ```
2. Switch to the FSMC directory
   ```
   C:>CHDIR \FSMC        [RETURN/ENTER]
   ```
3. Insert the FSMC program diskette into drive A
   Switch to A: drive
   ```
   C:>A:                 [RETURN/ENTER]
   ```
4. Start the system
   ```
   A:>FSMC               [RETURN/ENTER]
   ```
5. When asked "Do you have a hard drive [Y/N] ?", enter
   ```
   Y                     [RETURN/ENTER]
   ```
6. Enter a valid hard disk name
   ```
   C:                    [RETURN/ENTER]
   ```

The installation will then proceed. After the installation is completed, the main menu will appear. The C: drive will now be your current drive. Return the program diskettes to the manual for future installations.

2.3 INSTALLING FSMC MORE THAN ONCE

The FSMC system has no limit to the number of times it may be installed, however the installation procedures will appear automatically only the first time. To install the system more than once, select option "5-Utilities" from the main menu and select "Install the FSMC system". You will then be able to install the system as described above.

Note: Installation from option "5-Utilities" will only work from the floppy disks. It will not work from a hard disk because the FSMC copy of the program has already been installed to the hard disk drive.

3.0 GETTING STARTED

If the FSMC system has not yet been installed, proceed as described in the section entitled "Installing the System."

For a two floppy system, insert disk A into drive A and disk B into drive B and enter

```
A:>FSMC
```

For a hard drive system, change the current directory so that the FSMC is accessible, then enter

```
FSMC
```

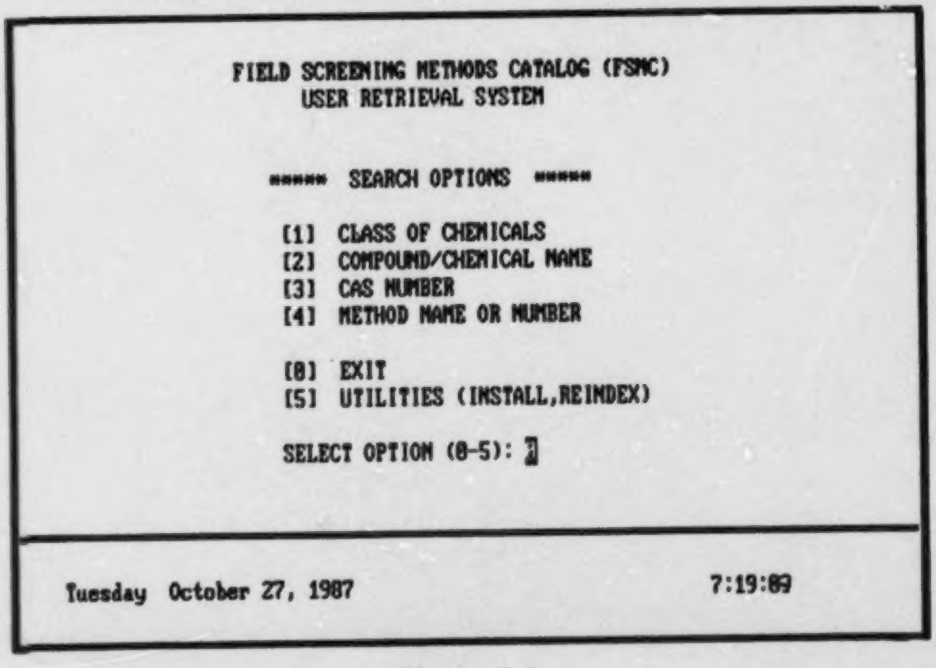
From this point on, all instructions are the same for both computer configurations.

3.1 SEARCHING THE DATA BASE

The data base may be searched in several ways. The user may enter a substance name or CAS number if a method for a specific chemical is desired, by chemical group (e.g. volatile organics), or by method name. The user's options are displayed and described in Figure 3-1. More detailed descriptions are included below.

The FSMC system provides several search options depending upon the information known about the substances and area to be sampled. The sequence of the search routines are summarized as follows:

1. Select an option in order to enter the chemical name, group, CAS number, or method name or number.
2. Select each matrix that must be analyzed with this field method by entering a "Y" after each matrix name.
3. Enter the detection limit to constrain the search to only those methods capable of analyzing below or equal to this limit for each matrix selected. Enter "0.0" if no limit is desired.
4. The default units of measure may be changed. Valid units of measure for the detection limit are PPM (parts per million) and PPB (parts per billion).
5. The system will respond by informing the user how many methods were found in this search. If there was at least one, a display/print menu will display the options of viewing the method descriptions.

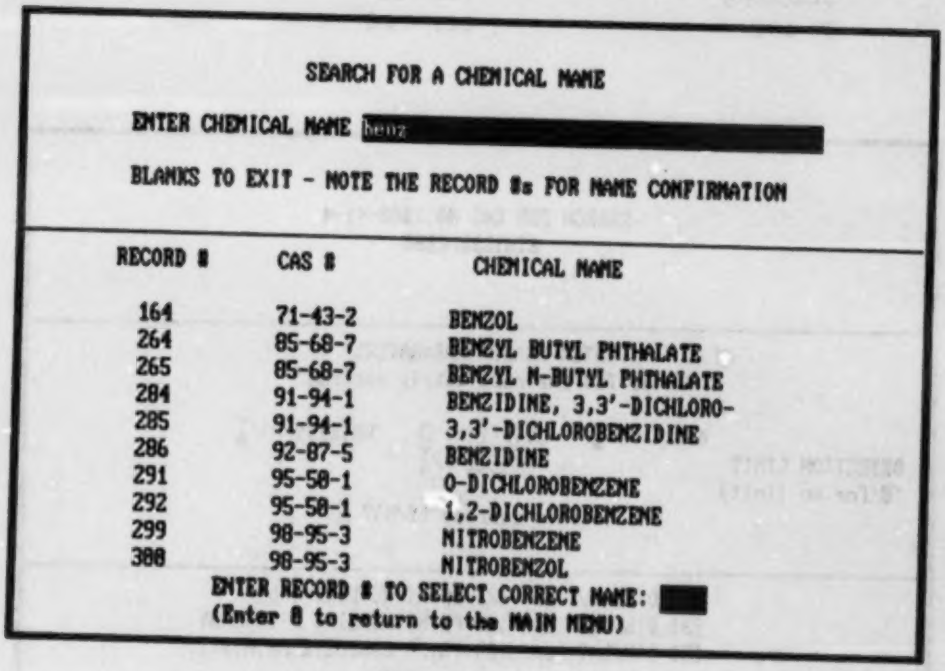


```
          FIELD SCREENING METHODS CATALOG (FSMC)
                USER RETRIEVAL SYSTEM

                ***** SEARCH OPTIONS *****

                [1]  CLASS OF CHEMICALS
                [2]  COMPOUND/CHEMICAL NAME
                [3]  CAS NUMBER
                [4]  METHOD NAME OR NUMBER

                [0]  EXIT
                [5]  UTILITIES (INSTALL,REINDEX)

               SELECT OPTION (0-5): 

Tuesday  October 27, 1987                           7:19:09
```

Figure 3-1

3.1.1 SEARCHING BY CHEMICAL NAME

The user may enter a specific chemical name. If the full name is not available, enter what you know is correct. The search is designed to look for an embedded character string within a chemical or method name. The system will match on what the user has entered if that character string is found anywhere in the name, display what was found, and then permit the user to select the one desired. See Figure 3-2.



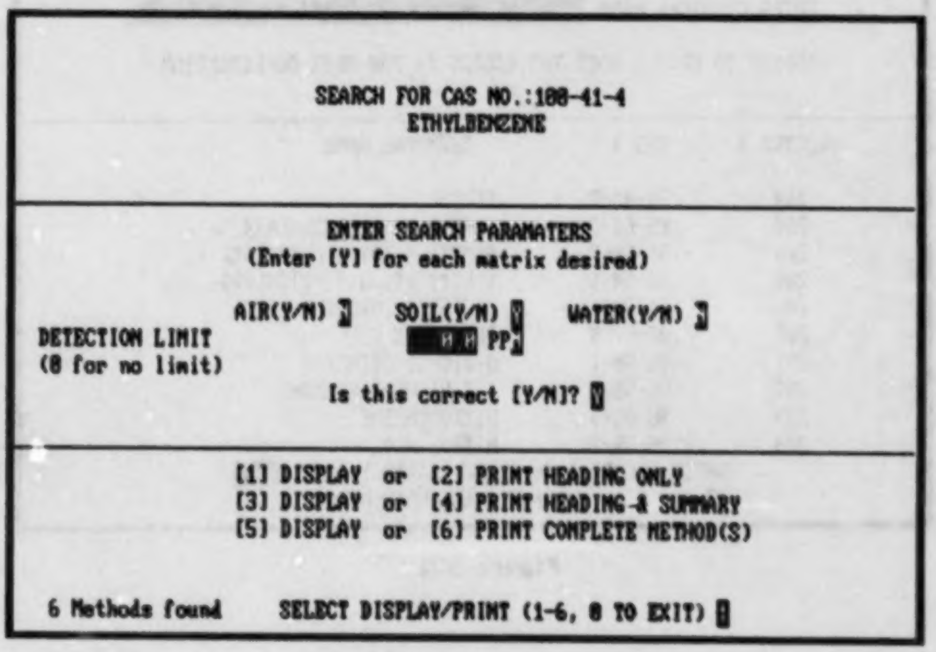
```
                    SEARCH FOR A CHEMICAL NAME

        ENTER CHEMICAL NAME benz

        BLANKS TO EXIT - NOTE THE RECORD #s FOR NAME CONFIRMATION

      RECORD #      CAS #          CHEMICAL NAME

        164        71-43-2        BENZOL
        264        85-68-7        BENZYL BUTYL PHTHALATE
        265        85-68-7        BENZYL N-BUTYL PHTHALATE
        284        91-94-1        BENZIDINE, 3,3'-DICHLORO-
        285        91-94-1        3,3'-DICHLOROBENZIDINE
        286        92-87-5        BENZIDINE
        291        95-50-1        O-DICHLOROBENZENE
        292        95-50-1        1,2-DICHLOROBENZENE
        299        98-95-3        NITROBENZENE
        300        98-95-3        NITROBENZOL
              ENTER RECORD # TO SELECT CORRECT NAME:
              (Enter 0 to return to the MAIN MENU)
```

Figure 3-2

3.1.2 SEARCHING BY CHEMICAL ABSTRACT SERVICES (CAS) NUMBER

The user must enter a Chemical Abstract Services (CAS) Number, including the dashes, and not including preceding zeros. The system will respond by displaying all chemical synonyms with that CAS number so that the user can verify the entry. One chemical name is then selected to continue the search. See Figure 3-3.

Valid CAS Numbers	Invalid CAS Numbers
7440-43-9	7429905
71-43-2	0057-74-9

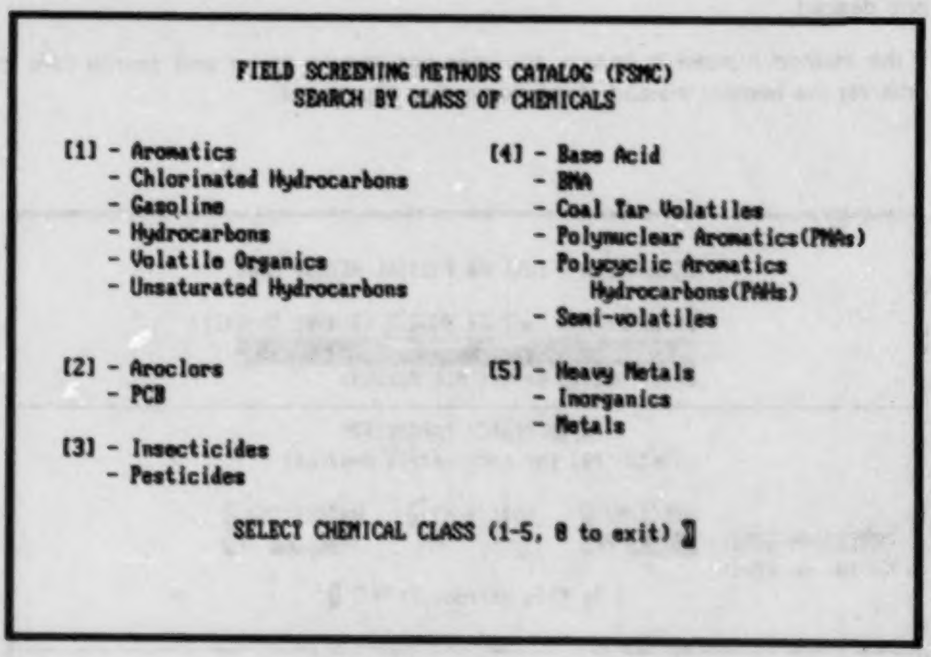


```
                     SEARCH FOR CAS NO.:100-41-4
                            ETHYLBENZENE

                      ENTER SEARCH PARAMATERS
                  (Enter [Y] for each matrix desired)

                 AIR(Y/N) N    SOIL(Y/N) Y    WATER(Y/N) N
DETECTION LIMIT                   0.0 PP
(0 for no limit)
                      Is this correct [Y/N]? Y

                 [1] DISPLAY  or  [2] PRINT HEADING ONLY
                 [3] DISPLAY  or  [4] PRINT HEADING-& SUMMARY
                 [5] DISPLAY  or  [6] PRINT COMPLETE METHOD(S)

 6 Methods found     SELECT DISPLAY/PRINT (1-6, 0 TO EXIT)
```

Figure 3-3

3.1.3 SEARCHING BY CHEMICAL CLASS

The user may request a search by one of five chemical class. These classes are shown in Figure 3-4.



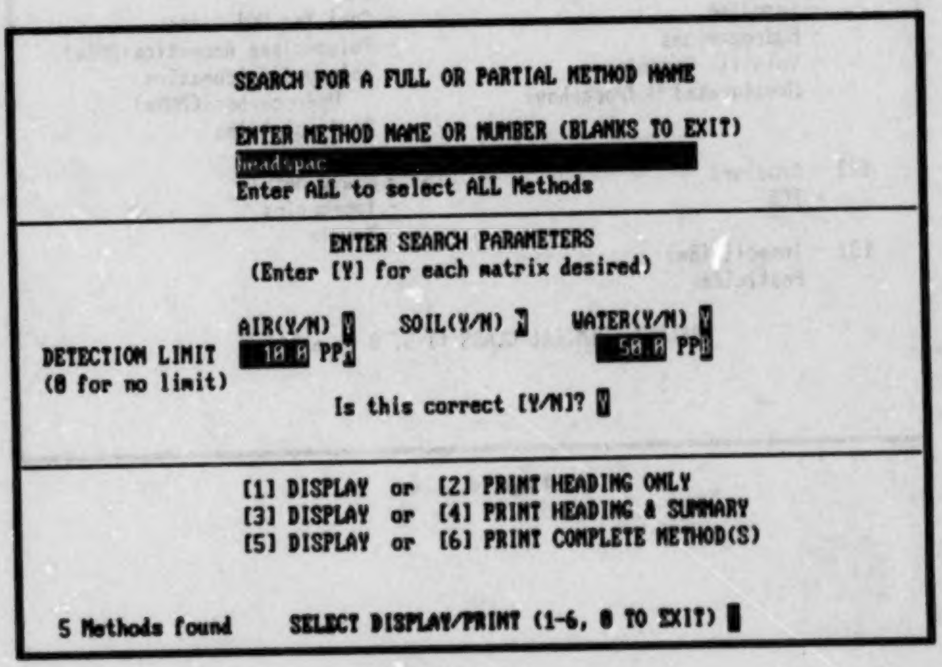
```
                  FIELD SCREENING METHODS CATALOG (FSMC)
                      SEARCH BY CLASS OF CHEMICALS

  [1] - Aromatics                         [4] - Base Acid
      - Chlorinated Hydrocarbons              - BNA
      - Gasoline                              - Coal Tar Volatiles
      - Hydrocarbons                          - Polynuclear Aromatics(PNAs)
      - Volatile Organics                     - Polycyclic Aromatics
      - Unsaturated Hydrocarbons                  Hydrocarbons(PAHs)
                                              - Semi-volatiles

  [2] - Aroclors                          [5] - Heavy Metals
      - PCB                                   - Inorganics
                                              - Metals
  [3] - Insecticides
      - Pesticides

                 SELECT CHEMICAL CLASS (1-5, 8 to exit) ▮
```

Figure 3-4

3.1.4 SEARCHING BY METHOD NAME OR NUMBER

The user may enter a specific method name. If the full name is not known, enter a character string that you know is correct. The search is designed to look for an embedded character string within a method name. The system will match on what the user has entered, display what was found, and then permit the user to select the one desired.

If the method number is known, the user can reduce typing and search time by entering the number instead of the name. See Figure 3-5.



```
                SEARCH FOR A FULL OR PARTIAL METHOD NAME

                ENTER METHOD NAME OR NUMBER (BLANKS TO EXIT)
                headspac
                Enter ALL to select ALL Methods
--------------------------------------------------------------------
                       ENTER SEARCH PARAMETERS
                 (Enter [Y] for each matrix desired)

                AIR(Y/N) Y     SOIL(Y/N) N    WATER(Y/N) Y
DETECTION LIMIT   10.0 PPM                       50.0 PPB
(0 for no limit)
                       Is this correct [Y/N]? Y
--------------------------------------------------------------------
                [1] DISPLAY  or  [2] PRINT HEADING ONLY
                [3] DISPLAY  or  [4] PRINT HEADING & SUMMARY
                [5] DISPLAY  or  [6] PRINT COMPLETE METHOD(S)

 5 Methods found     SELECT DISPLAY/PRINT (1-6, 0 TO EXIT)
```

Figure 3-5

3.1.5 SEARCHING BY THE SAMPLE MATRIX

The user may select specific matrices (air, soil and water) in order to constrain the search. The user must enter a "Y" for each matrix desired. The middle portion of Figure 3-5 displays the matrix options.

3.1.6 SEARCHING BY DETECTION LIMIT VALUES AND UNITS

If the user requires a certain detection limit or "sensitivity" level on which to search a method, the user may enter a value and units for each matrix selected. Only the methods that are valid at that detection limit or less will be selected. If a "0.0" is entered for any matrix, all methods for that matrix will be selected, regardless of the detection limit. The default units of measure may be changed by the user. Valid units of measure for the detection limit are PPM (parts per million) and PPB (parts per billion).

The middle portion of Figure 3-5 displays the detection limit option. The example shows a detection limit minimum for air (10.0 PPM) and for water (50.0 PPB).

4.0 DISPLAYING OR PRINTING REPORTS

If a search has been successful and one or more methods have been selected, a DISPLAY/PRINT menu presents the option to display the method(s) on the screen or to print them on an attached printer. This menu is shown in the bottom portion of Figure 3-5 and summarized in the following table.

Option Number	*Result*
1	Display Method Headings Only
2	Display Method Heading and Summary
3	Display Complete Method Listing
4	Print Method Headings Only
5	Print Method Heading and Summary
6	Print Complete Method Listing

After selecting one or more methods to print or display, the user may elect to print the detection limit table for each method. These tables will include all the substances for which each method may be applied. When the printing is complete, the DISPLAY/PRINT menu reappears. At this point you can choose to repeat the DISPLAY/PRINT process or exit to the main menu and conduct another search.

When a user selects options 3, or 6, methods selected for display or printing will produce fully detailed method descriptions. In order to view or print Headings only or Headings and Summaries, choose option 1 or 4 and 2 or 5 respectively.

5.0 FSMC SYSTEM REQUIREMENTS

5.1 SOFTWARE CHARACTERISTICS

The two sided 5 1/4 inch diskettes are the only software required to run the FSMC system. Users are encouraged to make backup copies of these two diskettes. Although users may freely copy the system diskettes, only users of registered copies will receive software and documentation revisions. To obtain additional copies of the FSMC system, contact the FSMC Coordinator at the address provided in the Executive Summary and Appendix C of this manual.

The FSMC system was developed by CDM Federal Programs Corporation using dBASE III Plus version 1.1, a product of Ashton-Tate Inc. The source code was compiled using Clipper, a product of Nantucket Software, Inc. and will run on most IBM compatible computers under MS-DOS. The FSMC system does not require a copy of dBASE III Plus to run, and will execute faster than if it were uncompiled running under dBASE III.

5.2 HARDWARE REQUIREMENTS

The following are the minimal computer hardware requirements needed to run the FSMC system. The system has been tested on Compaq DeskPro-386, Compaq Portable-2, Telex PC, IBM PC clones, and Tandon machines.

- IBM compatible computer with 512K of available RAM, a monochrome or color monitor.
- Two floppy disk drives or one floppy disk drive and one hard disk drive.
- A printer if printed reports are desired.

APPENDIX A

FIELD METHOD LISTING

LISTING OF METHODS

PRIMARY METHODS

FM-1 Field Atomic Absorption Analysis 1
FM-2 X-Ray Fluorescence in Laboratory for Heavy Metals 4
FM-3 X-Ray Fluorescence for Heavy Metals (On-site) 6
FM-4 Air Monitoring for Volatile Organic Compounds Using Programmed Thermal Desorption and GC 8
FM-5 Volatile Organic Compound Analysis Using GC with Automated Headspace Sampler 10
FM-6 Headspace Technique Using an Ion Detector for VOC Analysis. 12
FM-7 Headspace Technique Using an OVA for VOCs. 14
FM-8 Headspace Analysis Using HNU for Total Volatile Organics... 16
FM-9 Headspace Technique Using a Mobile GC for VOCs 18
FM-10 Passive Soil Gas Sampling Using Industrial Hygiene Samplers 20
FM-11 Soil Gas Sampling Using Mini-Barrel Sampler 22
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FM-D4 Immunoassays for Trace Organic Analysis 62
FM-D5 Use of Fiber Optic Sensors in Environmental Monitoring 64

Method FM-1: FIELD ATOMIC ABSORPTION ANALYSIS

Summary: Atomic absorption (AA) spectroscopy for metals analysis in field laboratory with the minimum detectable concentrations commonly below 1 ug/kg for metals such as Au, Ag, Cu, Cd, and Pb. Tungsten furnace requires a maximum of only 500 W of power and can be run off a portable generator.

Method description: Low mass tungsten furnace is used to atomize samples, making power requirements compatible with field lab use. Dry-ash-atomize timing and temperature controls must be se: for element being analyzed; final ashing temperature is adjustable between 1000°C and 1000°C; atomizing temperature is variable to 2500°C. Sample is prepared by procedures similar to those used in other AA techniques. Place 10 ul liquid sample by pipette onto atomizer. Analysis results are displayed in about 90 seconds.

Applications: Mobile field analysis for trace metals.

Limitations: Requires 110/220 volt electrical connection. Sample preparation required.

Instrumentation used: Scintrix AAZ-2 Zeeman Modulated Atomic Absorption Spectrophotometer.

PERFORMANCE SPECIFICATION

Sensitivity (Detection Limits): 0.1 mg/kg for most metals. Detection limits are obtained by taking 3 times the standard deviation from 14 determinations on a standard.

Selectivity: Zeeman technique used to reduce background interference.

Accuracy: Not generally available. Whenever possible, comparative results are presented in the user's Appliction Manual.

Precision/Repeatability: In general better than 10%. Measurements are based on 14 determinations of a metal standard which lies in the middle of the linear working range. For example, the RSD for 8 ng/ml copper was 3%, for 1.0 ng/ml of cadmium was 8% and for 20 ng/ml lead was 7%.

Comments: Portable generator can be used for power. Argon gas is used for cooling the furnace (eliminating need for water) and as a purge gas. Argon-hydrogen and helium are also used as purge gases based on specific applications. For each element being analyzed, a specific cathode lamp is required.

USE

Location Used: Field experience mainly related to mining, beginning to be used in hazardous waste work.

EPA Site Number (CERCLIS): Not Applicable.

Matrix: Water and Soil.

Preparation, Maintenance and Cleanup: Monochromator adjustment required to analyze for each element. Filament replacement in the furnace required after "hundreds" of firings. Argon gas used for flushing; 75 kg tank replacement in about 200 hours.

Analysis Time: 2 minutes/sample after sample preparation.

Capital Cost: $20,000 - $30,000.

Calibration: Three (3) standard solutions covering the entire working concentration range of the element being analyzed are used and prepared fresh daily. The standard solutions and a blank is run at the start of each day, and one standard is used to verify calibration three times a day.

Comments: Easily set up for field work in mobile or stationary field laboratory. Zeeman correction compensates somewhat for design compromise in optics and furnace required for portability. Hazardous waste site experience is limited.

Protocol Available:
Yes

SOURCE

Technical Contact: Dr. John D. Kinrade

Affiliation: Scintrex Limited

Telephone: (416) 669-2280

Prepared: 09/29/87

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Becker, D.L., and Carter, M.H., "Equipment Available for Sample Screening and On-Site Measurements, Appendices", TDD-HQ-8311-04, U.S. EPA, May 30, 1984 (Draft)

Kinrade J.D. et. al., "Applications Manual for the Scintrex AAZ-2 Zeeman Modulated Absorption Spectrophotometer" Scintrex Limited, Concord, Ontario, Canada October 1986

METHOD FM-2: X-RAY FLUORESCENCE (XRF) IN LABORATORY FOR HEAVY METALS

SUMMARY: Rapid screening of most metals (46) in soil and water in field laboratory to minimum of 20 mg/kg in soil. Conventional methods have better sensitivity and precision. Simultaneous detection (of the 18 elements analyzed for) is one of the greatest advantages of the system.

METHOD DESCRIPTION: Uses a flux of high energy x-rays to bombard sample causing elements in sample to emit characteristic wavelengths. The instrument separates the elements' wavelengths into a spectrum. Concentration of elements present is directly proportional to energies being produced. Technique used to screen soil and water samples. Soil sample preparation includes drying sample and grinding to a fine powder. Aqueous sample preparation includes concentrating the metallic

cations by filtering through strong acid ion exchange paper. Sample pH must be below 2 to ensure that metal ions are in cationic form. When anionic forms such as arsenate, etc. are present, base ion exchange is required. Region VIII method uses portable XRF analyzer which offers less sensitivity and detects fewer metals than this method.

APPLICATION: Rapid screening in laboratory for chromium, barium, cobalt, silver, arsenic, antimony, selenium, thallium, mercury, tin, cadium, lead, copper, nickel, zinc, manganese, iron, and vanadium.

LIMITATIONS: Does not have sensitivity or precision of atomic absorption or other conventional methods. Lithium, beryllium, aluminum, and boron not detected using this method.

INSTRUMENTATION USED: Kevex 7000 X-Ray Fluorescence Spectrometer.

PERFORMANCE SPECIFICATION

DETECTION LIMIT: Is element specific. For critical elements such as lead, it is 20 mg/kg in soil and 600 ug/l in water. For 18 elements tested, the range was from 20 to 50 mg/kg in soil and 100 to 600 ug/l in water.

SELECTIVITY: Elements may be identified by looking at various emission x-rays (i.e., K-alpha, K-beta, etc.). Spectra are stored on computer disc for later printout and direct identification of each element.

ACCURACY: Four samples analyzed for lead by CLP had values of 80, 180, 130 and 910 mg/kg. The range of values for the same samples analyzed by XRF were 100-300, 100-200, 95-120 and 800-900 mg/kg, respectively.

PRECISION/REPEATABILITY: Duplicate samples show good repeatability.

COMMENTS: XRF is non-destructive; samples can be stored for future reference after analysis.

USE

LOCATION USED: Sudbery, MA; 1985 (Non-CERCLA).

EPA SITE NUMBER (CERCLIS): Non-CERCLA

MATRIX: Soil and Water.

PREPARATION, MAINTENANCE AND CLEANUP: Soil samples (dried, 60 mesh-screened) are placed directly into sample cup; aqueous samples are ion exchanged by passing through a resin-coated filter paper. XRF spectrometer must be set up and programmed. Maintenance of the spectrometer includes checking probe for cleanliness and dryness and checking source decay. Standards are prepared using 1,000 mg/kg AA standard solutions for Ag, Ba, Mn, Ni, Sn, Zn, Se and Pb. Standards can be prepared separately or as multi-element mixtures and can be used up to 5 months.

ANALYSIS TIME: 10-30 minutes for sample preparation. Analysis time is less than 10 minutes.

CAPITAL COSTS: $80,000.00

CALIBRATION: Standards required at concentrations of 1000, 500, 250, and 125 mg/kg for soil and 2, 1, 0.5 and 0.25 ug/l plus a blank for water samples. Run all standards at beginning of each day and run a set every fourth hour of analysis or after all samples have been analyzed, whichever is more frequent. Additional standards must be prepared and used to cover the entire working range of required analyses.

COMMENTS: Spectrum displayed on video screen and stored in computer disk. Used routinely in Region I. Sample quantity needed for analysis is 1g of soil or 40 ml for water.

PROTOCOL AVAILABLE: Yes.

SOURCE

TECHNICAL CONTACT:	Dr. Thomas Spittler
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PREPARED:	April 7, 1987

BIBLIOGRAPHY

Furst, G.A., Spittler, T. and Tillinghust, V., "Screening For Metals at Hazardous Waste Sites: A Rapid Cost-Effective Technique Using X-Ray Fluorescence," Management of Uncontrolled Hazardous Waste Sites, Washington, D.C. November 4-6, 1985.

METHOD FM-3: X-RAY FLUORESCENCE (XRF) FOR HEAVY METALS (ON-SITE)

SUMMARY: Good for on-site screening of some metals in soil to minimum of 15 mg/kg. Sample matrix may cause accuracy problems. Uses portable XRF analyzer.

METHOD DESCRIPTION: Uses flux of high energy X-rays to bombard sample, causing elements in sample to emit energy at characteristic wavelengths. Instrument separates wavelengths produced into a spectrum that contains energy peaks characteristic of elements present. Concentration of each element present is directly proportional to the intensity of energy produced for that element. This technique has been used to screen soil and sediment samples for concentrations of lead, zinc, copper, arsenic, iron, and chromium. Sample preparation includes drying a 5-gram sample and grinding it to a fine powder. The XRF analyzer must be programmed and calibrated before samples are screened.

APPLICATION: Rapid on-site screening for lead, zinc, copper, arsenic, iron, and chromium

LIMITATIONS: Method does not have the sensitivity or precision of atomic absorption or other conventional methods. Cadmium, manganese, barium, and mercury may be detected, but only at high concentrations due to spectral overlap of other elements. Sample matrix effect may cause significant accuracy problems and can never be eliminated fully. (Matrix effects include non-homogeneity, surface conditions, and spectral interferences).

INSTRUMENTATION USED: XRF 840 analyzer electronic unit; HEPS sample probe, either Cm-244 or Am-241 radioisotope, or both.

PERFORMANCE SPECIFICATION

DETECTION LIMIT: 15 mg/kg for arsenic, to 140 mg/kg for iron but dependent on site-specific matrix and calibration techniques.

SELECTIVITY: Elements may be identified by looking at various emission X-rays (i.e., K-alpha, K-beta).

ACCURACY: Student's t-test and Wilcoxin's test show agreement between XRF and CLP data at a 95% confidence level for As, Cu, Pb, Zn, and Fe. (Number of samples ranged from 26-45.)

PRECISION/REPEATABILITY: Coefficient of variation = ± 20% at detection limit and ± 5% at higher values for all elements tested.

COMMENTS: Detection limit is directly related to the total number of x-rays counted and the number of x-rays due to interferences and background. Average RPD is about 27%; 18% may be due to sample non-homogeneity, 6% to instrument error and 3% due to the grinding process. Laboratory XRF analyzer offers better sensitivity performance.

USE

LOCATION USED: Smuggler Mountain Site, Aspen, Colorado, 1985

EPA SITE NUMBER (CERCLIS): COD980806277

MATRIX: Soil

PREPARATION, MAINTENANCE AND CLEANUP: The XRF analyzer must be set up and programmed. Maintenance includes checking the probe for cleanliness and dryness and checking for source decay.

ANALYSIS TIME: 10-30 minutes for sample preparation, depending on moisture content of the soil. Actual analysis averages 5 minutes per sample.

CAPITAL COSTS: $50,000

CALIBRATION: Sample calibration includes measurements of pure element calibrator standards, measurements of site specific samples with known analyte concentrations, input of calibration standard concentrations, and calculation of calibration coefficients. Midpoint standards should be rechecked after five samples. If deviation of standards is greater than 3%, recalibrate instrument.

COMMENTS: XRF is non-destructive; the samples can therefore be stored for future reference after analysis. Personnel training is required. Data reporting format is either direct LCD read-out or paper printout if interfaced with a printer.

PROTOCOL AVAILABLE: Yes

SOURCE

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PREPARED: April 6, 1987

BIBLIOGRAPHY

Chappell, R.W., Davis, A.O. and Clsen, R.L., "Portable X-Ray Fluorescence as a Screening Tool for Analysis of Heavy Metals in Soils and Mine Waste." Management of Uncontrolled Hazardous Waste Sites, Washington, D.C. December 1-3, 1986.

Chappell, R.W. and Olsen, R.L., EPA Memorandum, "XRF Field Analysis of Smuggler Mountain Soil Samples," January 13, 1986.

Columbia Scientific Industries Corporation, "Operting Instructions X-MET 840 Portable XRF Analyzer", March 1985.

METHOD FM-4: AIR MONITORING FOR VOLATILE ORGANIC COMPOUNDS USING PROGRAMMED THERMAL DESORBER (PTD) AND GC

SUMMARY: Air monitoring of volatile organics as a time-weighted average to ug/l range. Time consuming. Requires field laboratory.

METHOD DESCRIPTION: Uses a field sampling pump, programmed thermal desorber, and a GC to evaluate volatile organic contaminants in air samples as a time-weighted average. Identification and quantitation is done by comparing peak retention times and heights of peaks with standards. Sample acquisition involves collecting an adequate sample (usually 10-30 liters) of ambient air using a Tenax or activated carbon tube attached to a pump. The sample is thermally desorbed in the PTD. Two samples are withdrawn from the PTD and analyzed on the GC. The first sample is usually a small volume "preliminary" sample, and the second is a larger volume sample, based on results from the first.

APPLICATION: Air monitoring as a time-weighted average for low molecular weight volatile organic compounds; aromatics; unsaturated hydrocarbons; chlorinated hydrocarbons; ketones; alcohols, etc.

LIMITATIONS: Time consuming; samples take 4-8 hours to collect. Desorption efficiency is less than 100%. Pre-packed Tenax tubes may be contaminated. Packing own tubes is recommended but requires laboratory and chemist.

INSTRUMENTATION USED: Century Systems Programmed Thermal Desorber, Model 132 A.

Century Systems (Foxboro) Organic Vapor Analyzer or other comparable GC or GC/MS (e.g., Photovac, Finnigan, etc.).

PERFORMANCE SPECIFICATION

DETECTION LIMIT: 1.0 ug/l using the Photovac or Finnigan GC/MS for most volatile organics; or 500 ug/l using Foxboro OVA for most volatile organics.

SELECTIVITY: Good separation of peaks.

ACCURACY: 99% recovery of eleven spiked vinyl chloride samples using freshly packed coconut charcoal tubes; lower recovery in other tests possibly related to spiking method.

PRECISION/REPEATABILITY: Standard deviation of spiked vinyl chloride samples was approximately 10%.

COMMENT: Recoveries varied according to type and condition of carbon tube used and number of components in sample. With 8 components, recovery ranged from 3-106%; for 4 components, recovery range was 63-135%.

USE

LOCATION USED: Has been used at 30-50 locations in New England and elsewhere.

EPA SITE NUMBER (CERCLIS): Not Available

MATRIX: Air

PREPARATION, MAINTENANCE AND CLEANUP: Tubes are prepared by thermally desorbing in PTD and cleaned to background levels (less than 1 ug/l). If tubes are dirty or contaminated they may have to be cleaned and repacked or replaced.

ANALYSIS TIME: 4-8 hours for sample collection; 5 minutes desorbing; 5 minutes analysis time.

CAPITAL COSTS: $5,990 for Model 132A PTD; $5,200-$6,325 for OVA

CALIBRATION: Calibration determined by peak heights and retention times for standards. Run standards as for GC or GC/MS.

COMMENTS: The interpretation of results requires a trained chemist. Method is used on average 2-3 times per year in Region I.

PROTOCOL AVAILABLE: Yes.

SOURCE

TECHNICAL CONTACT: Dr. Thomas Spittler

AFFILIATION: U.S. EPA Region I Laboratory

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PREPARED: April 7, 1987

BIBLIOGRAPHY

Spittler, T.M., Siscanaw, R.J. and Lataille, M.M., "Correlation between Field GC Measurement of Volatile Organics and Laboratory Confirmation of Collected Field Samples Using the GC/MS." National Conference on Management of Uncontrolled Hazardous Waste Sites, November 29 - December 1, 1982, Washington, D.C.

Chapman H. and Clay P., Field Investigation Team (FIT) Screening Methods and Mobile Laboratories Complementary to Contract Laboratory Program (CLP). TDD-HQ-8507-01, October 17, 1986.

METHOD FM-5: VOLATILE ORGANIC COMPOUND (VOC) ANALYSIS USING GC WITH AUTOMATED HEADSPACE SAMPLER

SUMMARY: Rapid determination of VOCs to 10 ug/l in water and 10 ug/kg in soil. Requires field laboratory. Results are tentative. Method yet to be used in field.

METHOD DESCRIPTION: Requires a field laboratory with an automated headspace sampler interfaced to a GC equipped with a PID and ECD in series to screen VOCs in soil and water. Identification and quantitation by comparison of standard peak retention times and peak areas with sample. Method for water consists of transferring 1 ml of water sample to a 3 ml reaction vial. For soil, method consists of transferring 1 gram of sample to 3 ml reaction vial. Add 1 ml of surrogate standard to each sample. Set headspace sampler temperature at 60oC. Load sampler and analyze samples.

APPLICATION: Good for most VOCs to low ug/kg ranges; halogenated hydrocarbons, chlorinated hydrocarbons, aromatics, etc.

LIMITATIONS: Complex samples give co-eluting peaks. Results, especially for soil, are semi-quantitative. Method has not been used in the field.

INSTRUMENTATION USED: Perkin-Elmer 2000 with HNU PID detector and Tracor Hall detector. Hewlett Packard Model 19395A automated headspace sampler.

PERFORMANCE SPECIFICATION

DETECTION LIMIT: 10 ug/l for most VOCs in water. 20 ug/l for some halogenated aromatics. 10 ug/kg for most VOCs in soil.

SELECTIVITY: Peaks are separate, occasional overlapping peaks.

ACCURACY: At low concentrations (10-50 ug/l) good correlation to GC/MS data. At higher levels, there is a discrepancy between data sources by a factor of two. Bias ranged from high to low with increasing concentrations.

PRECISION/REPEATABILITY: Not available.

COMMENTS: This method was developed by Region IV and until recently was not used as a quantitative method.

USE

LOCATION USED: Not used in field.

EPA SITE NUMBER (CERCLIS): Not applicable

MATRIX: Air (Headspace above soil, sediment, or water)

PREPARATION, MAINTENANCE AND CLEANUP: Column and detector cleaning and reconditioning almost nonexistent because of the nature of headspace analysis. Samples must equilibrate in sampler 1 hour before analysis.

ANALYSIS TIME: 30-40 min/sample

CAPITAL COST: Approximately $20,000 for Perkin-Elmer GC with PID and ECD. Approximately $8,000 for Hewlett Packard headspace sampler.

CALIBRATION: 3-point standard calibration curve necessary to accurately quantify compounds detected by GC. Single point calibration is adequate for semiquantitative data. Standard is known quantity of organic vapor in equilibrium with air with an aqueous organic solution.

COMMENTS: The interpretation of results requires a trained chemist. This method has been used in Region IV laboratory but has yet to be used in the field.

PROTOCOL AVAILABLE: Yes.

SOURCE

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PREPARED: April 16, 1987

BIBLIOGRAPHY

Screening Method for Volatile Organic Compounds, EPA Region IV Mobile Laboratory Protocol, January 1987.

METHOD FM-6: HEADSPACE TECHNIQUE USING AN ION DETECTOR FOR VOC ANALYSIS

SUMMARY: Analysis of soil, water, and air samples for identification and quantitation of most VOCs to ug/l range. The ion detector is a GC/MS system. Requires field laboratory. Soil results are semi-quantitative.

METHOD DESCRIPTION: Used to screen water, soil and sediment samples for volatile organic compounds (VOC) on an ion trap detector— a gas chromatograph/mass spectral (GC/MS) system — in a field laboratory. GC separates the compounds and introduces them into MS which provides positive identification of peaks. Quantitation is determined by peak areas on GC. Method involves collecting desired sample in a 40 ml vial, preparing sample (if soil) and sampling the vapor headspace above solution. It is then analyzed on ion trap detector. Air is screened directly by collecting sample and injecting it into GC for analysis.

APPLICATION: Good for most VOCs to 1 ug/l concentrations; unsaturated hydrocarbons, halogenated hydrocarbons, aromatics, etc. Mass spectrum good for more positive identification of compounds.

LIMITATIONS: Results for soil are semi-quantitative. High initial cost.

INSTRUMENTATION USED: Finnigan Ion Trap Detector (ITD) 700 series with IBM PC/XT

PERFORMANCE SPECIFICATION

DETECTION LIMIT: 1 ug/l for most VOCs

SELECTIVITY: Excellent identification capabilities even in the presence of co-eluting peak.

ACCURACY: Six-point calibration curve yielded 0.964 correlation coefficient.

PRECISION/REPEATABILITY: Six-point calibration curve showed 20% relative standard deviation.

COMMENT: Performance specifications were taken from headspace of water samples.

USE:

LOCATION USED: Adamstown, Maryland; Well Water Analysis, August 1986.

EPA ID NUMBER: Non-CERCLIS

MATRIX: Air (Headspace above soil, sediment, and water)

PREPARATION, MAINTENANCE AND CLEANUP: Columns and detector must be cleaned and reconditioned occasionally. Care must be taken not to saturate the column with concentrated samples.

ANALYSIS TIME: 10-20 minutes/sample

CAPITAL COST: Approximately $40,000 for ITD with IBM PC/XT

CALIBRATION: Standards necessary to identify and quantify compounds. The standard is a known quantity of an organic vapor, which is in equilibrium with either air or an aqueous or water-organic solution. Minimum of three points should be used to develop calibration curve.

COMMENTS: The interpretation of results requires a trained chemist. Method has seen limited use.

PROTOCOL AVAILABLE: No

SOURCE:

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PREPARED: April 23, 1987

BIBLIOGRAPHY

Chapman, H. and Clay, P., "Field Investigation Team (FIT) Screening Methods and Mobile Laboratories Complementary to Contract Laboratory Program (CLP)," TDD-HQ-8507-1, October 17, 1986, (Draft).

METHOD FM-7: HEADSPACE TECHNIQUE USING AN OVA (FID) — VOLATILE ORGANIC COMPOUNDS

SUMMARY: Rapid analysis of total VOCs or identification of individual components in soil, water, or air. OVA is portable. Soil results are semi-quantitative. Field instruments give better sensitivity.

METHOD DESCRIPTION: Used to screen water, soil, and sediment samples on an OVA— a portable GC equipped with a flame ionization detector. Method involves collecting samples in a 40 ml vial, preparing sample if it is soil or sediment, and sampling the vapor headspace above the aqueous solution. The vapor sample is analyzed by the OVA. Air may be screened directly by injecting it into the OVA. Identification and quantitation determined by comparing the peaks of standards to samples. A modification of this method involves placing the probe directly above the soil sample or inserting the probe directly into a shallow bore hole.

APPLICATION: Analysis for low molecular weight total volatile organic concentration or for identification of specific constituents with the use of proper standards.

LIMITATIONS: Measures volatile organics only. Highly volatile organics such as methane tend to skew analysis for total VOCs. Light VOCs (e.g., vinyl chloride) are rapidly eluted from column and are difficult to detect. Response given in methane equivalent. OVA operates at ambient temperatures; therefore for reproducibility, surrounding temperatures must remain constant.

INSTRUMENTATION USED: Century Systems (Foxboro) Organic Vapor Analyzer (Model 128).

PERFORMANCE SPECIFICATION

DETECTION LIMIT: 500 ug/l for most volatile organics.

SELECTIVITY: Early peaks tend to overlap. Later peaks easily identifiable.

ACCURACY: Calibration error (for benzene) ranged from 25% of value at detection limit (0.74 mg/kg) to 14% at higher concentrations (165 mg/kg).

PRECISION/REPEATABILITY: Coefficient of variance = 1-4% for five standard samples of benzene and carbon tetrachloride. Duplicate samples show good agreement.

COMMENT: Performance specifications are for air; results for soil/sediment semi-quantitative.

USE

LOCATION USED: Ottadi & Gross (Kingston Steel Drum); Kingston, NY 1980.

EPA SITE NUMBER (CERCLIS): NHD990717647

MATRIX: Air (Headspace above soil, sediment, and water)

PREPARATION, MAINTENANCE AND CLEANUP: Columns must be cleaned every 3 months. Recharge batteries after use.

ANALYSIS TIME: 20 samples per hour.

CAPITAL COSTS: $7,000.

CALIBRATION: Standards necessary to identify and quantify compounds detected by GC. The standards are run before screening field samples.

COMMENTS: The interpretation of results requires a trained chemist. The column can easily be saturated by concentrated samples, resulting in an inoperable unit. Method is routinely used in Region I.

PROTOCOL AVAILABLE: Yes

SOURCE

TECHNICAL CONTACT: Dr. Thomas Spittler
AFFILIATION: U.S. EPA Region I Laboratory
TELEPHONE: (617) 861-6700
PREPARED: April 7, 1987

BIBLIOGRAPHY

Quimby, J.M., Cibulskis, R.W. and Gruenfeld, M., "Evaluation and Use of a Portable Gas Chromatograph For Monitoring Hazardous Waste Sites." National Conference on Management of Uncontrolled Hazardous Waste Sites, November 29 - December 1, 1982, Washington, D.C.

METHOD FM-8: HEADSPACE ANALYSIS USING HNU (PID) FOR TOTAL VOLATILE ORGANICS

SUMMARY: Portable instrument that gives rapid analysis of total organic vapor concentrations in water, soil, and sediment to 100 ug/l benzene equivalent.

METHOD DESCRIPTION: Used to screen water, soil, and sediment samples for total organic vapor concentration. HNU is a portable photoionization detector that requires internal electronic calibration as well as calibration to a known standard. After sample is collected, it can be analyzed by inserting probe of HNU into headspace of jar. Alternative procedures to place probe directly above soil or insert probe into a shallow hole.

APPLICATION: Measures total organic vapor concentration. Response to VOC varies with probe used. Insensitive to methane. May detect unsaturated hydrocarbons, chlorinated hydrocarbons, aromatics, nitrogen and sulfur compounds, aldehydes, ketones, alcohols, acids, and others.

LIMITATIONS: Not able to identify individual compounds. Total response reported as benzene equivalent. High ambient humidity causes erratic responses (usually low).

INSTRUMENTATION USED: HNU Systems PI 101 Portable Photoionizer. Available probes include 9.5 ev, 10.2 ev and 11.7 ev.

PERFORMANCE SPECIFICATION

DETECTION LIMIT: 100 ug/l for most volatile organics. Linear operating range for most compounds is 100-60,000 ug/l. Useful range extends to 200,000 ug/l.

SELECTIVITY: Depends on probe.

ACCURACY: Not tested.

PRECISION/REPEATABILITY: 1% at Full Scale Deflection. Duplicate samples show good agreement.

COMMENTS: Three probes are available that vary in sensitivity to organic compounds. One probe (9.5 ev) detects aromatics and large molecules. The 10.7 ev probe detects the above compounds plus vinyl chloride, MEK, TCE and other 2-4 carbon compounds. The 11.7 ev probe detects the above compounds plus halocarbons, methanol, and other single carbon compounds.

USE:

LOCATION USED: Ottadi & Gross (Kingston Steel Drum); Kingston, NY, 1980.

EPA SITE NUMBER (CERCLIS): NHD990717647

MATRIX: Air (Headspace above soil, sediment, and water)

PREPARATION, MAINTENANCE AND CLEANUP: Recharge battery after use. Clean light source window every few weeks.

ANALYSIS TIME: Response given in less than 5 seconds

COST: $5,000.00

CALIBRATION: Zero electronically at start of day. Check periodically. Use calibration gas prior to each use.

COMMENTS: Easy to train personnel in usage. Used fairly regularly.

PROTOCOL AVAILABLE: No.

SOURCE

TECHNICAL CONTACT: Dr. Thomas Spittler

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TELEPHONE: (617) 861-6700

PREPARED: April 13, 1987

BIBLIOGRAPHY

Becker, D.L. and Carter, M.H., "Equipment Available for Sampling and On-Site Measurements." U.S. EPA, Document Number HQ-8311-04, May 30, 1984.

METHOD FM-9: HEADSPACE TECHNIQUE USING A MOBILE GC FOR VOLATILE ORGANICS (VOCS)

SUMMARY: Analysis of soil, water, and air samples for identification and quantitation of most VOCs to 0.5 ug/l range. Soil results are semi-quantitative. Depending on GC used, is portable or requires field laboratory.

METHOD DESCRIPTION: Used to screen water, air, soil, and sediment samples on a GC with a PID, FID or ECD. Method involves collecting desired sample in a 40 ml vial, preparing sample if soil or sediment, and sampling and analyzing vapor headspace above aqueous solution. Air screened directly by collecting sample and injecting into GC for analysis. Identification and quantitation is determined by comparing peak retention times and areas of standard solution to samples.

APPLICATION: Good for most VOCs to low concentrations; halogenated methanes and ethanes, chlorinated hydrocarbons, aromatics, etc; arsine, phosphine, hydrogen sulfide, and carbon disulfide.

LIMITATIONS: GC and sample need to be at same temperature in an area free of organic vapor. For reproducibility, surrounding temperature must remain constant. Complex samples give co-eluting peaks. Identifications are considered tentative for soil.

INSTRUMENTATION REQUIRED: Photovac Model 10A10 equipped with FID and 4 foot SE-30 column; AID 511 equipped with FID or ECD and 3-foot SE-30 column; Shimadzu GC Mini-2 and GC Mini-3 with FID and 6-foot SP-1000 and AT-1000 column; HNU Model GC-301 with PID or FID and Standard SE-column.

PERFORMANCE SPECIFICATION

DETECTION LIMIT: 1 ug/l for aromatics; 40 ug/l for some chlorinated hydrocarbons.

SELECTIVITY: Peaks are separate, good selectivity.

ACCURACY: 27-32% RPD (Standard Deviation) (70 TCE and PCE samples).

PRECISION/REPEATABILITY: Duplicate samples show very good agreement.

COMMENTS: Results given for Photovac, the most common GC used. Others give similiar results.

USE

LOCATION USED: Norwalk Harbor, Conn. Winter 1981

EPA SITE NUMBER (CERCLIS): Non-CERCLA

MATRIX: Air and headspace above soil, sediment, and water.

PREPARATION, MAINTENANCE AND CLEANUP: Optional backflush valve allows for flushing of contaminants from column. Column conditioning done every third month by heating to 100ºC and flushing with helium.

ANALYSIS TIME: 10-20 minutes.

CAPITAL COSTS: Photovac: $14,000.00
AID 511: $5,020.00
Shimadzu: $4,100.00-$5,200.00
HNU-301: $6,350.00

CALIBRATION: Standard necessary to identify and quantify compounds detected by GC. Standard is a known quantity of organic vapor in equilibrium with air or an aqueous or organic solution. Standards run before screening field samples and after every 5 samples..

COMMENTS: The interpretation of results requires a trained chemist. The column can easily be saturated by concentrated samples, resulting in an inoperable unit. Used routinely.

PROTOCOL AVAILABLE: Yes

SOURCE

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PREPARED: April 7, 1987

BIBLIOGRAPHY

Chapman, H. and Clay, P., "Field Investigation Team (FIT) Screening Methods and Mobile Laboratories Complementary to Contract Laboratory Program (CLP)," TDD HQ-8507-01, October 17, 1986. (Draft).

Clark, A.E., Lataille, M. and Taylor, E.L., "The Use of a Portable PID Gas Chromatograph for Rapid Screening of Samples for Purgeable Organic Compounds in the Field and in the Lab," U.S. EPA Region I Laboratory, June 29, 1983.

Morin, S.O., "Development and Application of an Analytical Screening Program to Superfund Activities," Management of Uncontrolled Hazardous Waste Sites, Washington, D.C. November 4-6, 1985.

METHOD FM-10: PASSIVE SOIL GAS SAMPLING USING INDUSTRIAL HYGIENE SAMPLERS

SUMMARY: Passive sampling for volatile organics as a way to detect and estimate ground water contamination. Concentrations obtained by calculation. Site conditions affect results. Field laboratory or off-site analysis possible.

METHOD DESCRIPTION: Passive sampling using open, inverted 1-quart metal cans containing an activated carbon organic vapor monitor are buried at one-foot depths in area to be sampled, exposed for a time determined by sampling rate for target chemical and anticipated soil concentrations of target chemical (8 hours to 1 month), followed by solvent desorption in off-site or field laboratory and GC analysis (with ECD, PID or FID). Soil gas concentration of chemical by volume is calculated using results of analysis, sample exposure time, and sampling rate for chemical of interest.

APPLICATION: Assessment of VOC ground water contamination plume by soil gas sampling.

LIMITATIONS: Does not provide ground water contaminant concentrations directly; is an indirect method. Site conditions affect method.

INSTRUMENTATION USED: Activated carbon organic vapor monitors (#3500, 3M, St. Paul, MN). Various GCs and column configurations have been successfully used including the HP 5710 GC/ECD, the AID GC/ECD and the HNu 301 GC/FID/PID.

PERFORMANCE SPECIFICATION

DETECTION LIMIT: Depends on GC used, local conditions, and exposure time.

SELECTIVITY: Volatile organics; depends on GC method used.

ACCURACY: Correlation of 0.93 between this technique and grab samples obtained earlier; correlation of 0.79 with ground water monitoring results.

PRECISION/REPEATABILITY: Based on closely spaced samples. 12% RSD over 27'.

COMMENTS: Need to demonstrate, precision and correlation with ground water contamination at each site. Test precision using closely spaced samplers, and evaluate correlation with ground water data by regression analysis.

USE

LOCATION USED: Pittman Lateral, Henderson, NV (Numerous other locations).

EPA SITE NUMBER (CERCLIS): Non-CERCLA

MATRIX: Soil

PREPARATION, MAINTENANCE AND CLEANUP: Solvent desorption required before GC analysis.

ANALYSIS TIME: Sample collection time varies depending on analytical sensitivity, expected concentration of contaminant, etc. Sample processing involves solvent desorption (1/2 hour) and GC analysis (15-30 min/sample).

CAPITAL COST: Not available

CALIBRATION: Check validity of method under site conditions.

COMMENTS: One of several soil gas sampling methods that may be applicable under various circumstances. Clay layers and horizons with <5% air-filled porosity can reduce efficiency of soil gas sampling.

PROTOCOL AVAILABLE: Yes

SOURCE

TECHNICAL CONTACT: Henry B. Kerfoot

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PREPARED: May 6, 1987

BIBLIOGRAPHY

Kerfoot, H.B. & Mayer, C.L., "The Use of Industrial Hygiene Samplers for Soil-Gas Surveying," Ground Water Monitoring Review, Fall 1986, 74-78.

METHOD FM-11: SOIL GAS SAMPLING USING MINI-BARREL SAMPLER

SUMMARY: Method to collect soil gas samples when low concentration expected. Soil aliquots are directly removed and prepared. Headspace gas is withdrawn and injected into GC. Analysis can be performed using portable GC, field and/or off-site laboratories.

METHOD DESCRIPTION: A coring tool is used along with an auger or backhoe to collect the sample. Samples are forced into screw-cap vials by a sampling extruder. Deionized/distilled water is added to the vial, which is then heated, agitated, and samples are withdrawn for GC analysis.

APPLICATIONS: Suited for low-concentration contaminants with low vapor pressures or where sample contaminants need to be concentrated in a headspace to be within detection limits of the analytical instument.

LIMITATIONS:

INSTRUMENTATION USED: Mini-barrel borehole sampler and sample extruder.

PERFORMANCE SPECIFICATION

DETECTION LIMITS: Suited for low-concentration contaminants.

SELECTIVITY: Volatile organics.

ACCURACY: Not Known

PRECISION/REPEATABILITY: Not Known

COMMENTS: Clay layers and horizons with <5% air-filled porosity reduce effectiveness of soil gas sampling. Compare with other sampling techniques to determine applicability at each site.

USE

LOCATION USED: Not available

EPA SITE NUMBER (CERCLIS): Not applicable

MATRIX: Soil.

PREPARATION, MAINTENANCE AND CLEANUP: N/A

ANALYSIS TIME: N/A

CAPITAL COST: Not Available

PROTOCOL AVAILABLE: Yes.

SOURCE

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PREPARED: May 6, 1987

BIBLIOGRAPHY

Chapman, H., and Clay, P., Field Investigation Team (FIT) Screening Methods and Mobile Laboratories Complementary to Contract Laboratory Program (CLP), TDD-HQ-8507-01, October 17, 1986 (Draft).

METHOD FM-12: SOIL GAS SAMPLING USING A ONE-LITER SYRINGE

SUMMARY: Rapid sampling of soil gas for on-site analysis to determine contaminant plume; repeated sampling not possible once auger removed. GC analysis can be performed in a field laboratory.

METHOD DESCRIPTION: Sample is obtained as follows: drill hole using hollow-stem auger; lower a length of Teflon tubing attached to a 1-liter, gas-tight syringe to the desired sampling depth. Condition Teflon line by drawing a 500 ml air sample, detach and evacuate the syringe, reattach and draw 1-liter sample, which is injected into a purge and trap unit for concentration and GC analysis.

APPLICATION: Soil sampling for GC analysis to aid in contaminant plume delineation.

LIMITATIONS: Relatively long analysis time; no repeated sampling once auger is removed; variation in injection rate can lead to VOC breakthrough in the purge and trap unit.

INSTRUMENTATION USED: Sampling: 1-liter, gas-tight syringe, syringe pump. Analysis: GC with a purge and trap unit.

PERFORMANCE SPECIFICATION

DETECTION LIMIT: Sensitivity improved over other sampling techniques by sample concentration (0.2-0.02 ug/l for TCE).

SELECTIVITY: Volatile organics; depends on GC method used for analysis.

ACCURACY: High correlation with other sampling techniques: Correlation coefficients were 0.68 with direct injection auger; 0.88 with soil headspace; 0.87 with direct injection-stopper; 0.90 with Tenax tube.

PRECISION/REPEATABILITY: N/A

COMMENTS: Sensitivity depends on GC used; concentrating techniques overcome some field GC limitations. Clay layers and horizons with <5% air-filled porosity reduce effectiveness of soil gas sampling. Method should be tested at each site by comparing to results of other sampling techniques.

USE

LOCATION USED: Puget Sound, WA, 1986.

EPA SITE NUMBER (CERCLIS): Not Available

MATRIX: Soil.

PREPARATION, MAINTENANCE AND CLEANUP: Syringes and tubing must be cleaned (e.g., using vacuum heated syringe cleaner following sample injection); nitrogen gas flushing. Decontamination of auger & syringes required for reuse.

ANALYSIS TIME: Drilling borehole— 15-30 min; sample collection— 1 min; GC analysis— 30 minutes.

CAPITAL COSTS: Syringe ($30-40); pump($100-500); Both are reusable.
GC $4,100-$20,000,

CALIBRATION: N/A

COMMENTS: Simple sample collection. May perform better than Direct Injection - Auger sampling when most appropriate analytical equipment used. Allows adjustment of sampling area for further investigation based on immediate results.

PROTOCOL AVAILABLE: No.

SOURCE

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PREPARED:	May 6, 1987

BIBLIOGRAPHY

Jowise, P. P., Villnow, J. D., Gorelik, L. I., Ryding, J. M., "Comparative Analysis of Soil Gas Sampling Techniques," Management of Uncontrolled Hazardous Waste Sites, Washington, D.C., December 1-3, 1986, p. 193-199.

METHOD FM-13: SOIL GAS SAMPLING USING DIRECT INJECTION - STOPPER

SUMMARY: Sampling of VOCs in soil gas to determine ground water contaminant plume. Requires probe installation for equilibration and repeated sampling. Analysis can be performed using portable GC and/or field laboratory.

METHOD DESCRIPTION: A 12-17-foot borehole is drilled using a hollow-stem auger (8" O.D.). A sample probe is inserted, borehole walls are allowed to collapse as the auger is pulled. The hole is sealed with a bentonite-slurry plug, the probe is sealed with a stopper and screw cap. Samples are collected by syringe after a 2-day equilibration period and analyzed by a GC.

APPLICATION: Delineation of extent of contamination; long-term sampling of true soil gas concentrations.

LIMITATIONS: Probe installation required.

INSTRUMENTATION USED: Auger for drilling, gas-tight syringes for sampling; GC for analysis.

PERFORMANCE SPECIFICATION

DETECTION LIMIT: 0.2 ug/l for TCE in one study.

SELECTIVITY: Volatile organics; depends on GC method used.

ACCURACY: Results correlated well with results from other sampling methods at same locations: Correlation coefficients were 0.78— direct injection-auger; 0.87— one-liter method; 0.73— headspace method; 0.98— Tenax.

PRECISION/REPEATABILITY: Field variability - 33% RSD for 42 samples.

COMMENTS: Occasional leaks in stoppers increased variability. Water contamination accurately reflected; however, number of samples was small and correlation may be misleading. Sensitivity depends on GC used. Clay layers and horizons with <5% air-filled porosity reduce effectiveness of soil gas sampling. Compare to other sampling methods to assess applicability to a particular site.

USE

LOCATION USED: Puget Sound, WA, 1986.

EPA SITE NUMBER (CERCLIS): Not Available

MATRIX: Soil.

PREPARATION, MAINTENANCE AND CLEANUP: Determine appropriate drilling depth by use of test holes (need to drill below surface soils to bypass competing adsorption sinks) or by combined experience and assessment of site factors. Auger and syringe decontamination required. Periodic direct mixing of probe contents needed before sampling to provide equilibrium conditions along probe length. Minimal sample preparation required before analysis.

ANALYSIS TIME: 15-30 minutes to drill boreholes; 2 days for sample probe equilibration; 1 minute to collect sample.

CAPITAL COST: Syringes ($30-40), probes ($100) plus drilling equipment
GC: $4,100 - $20,000

CALIBRATION: Purge syringes with nitrogen and check for contamination by injecting carrier gas samples into GC. Periodic mixing of probe contents.

COMMENTS: Long-term changes in soil gas concentration will not be evident without mixing of probe contents.

PROTOCOL AVAILABLE: Yes.

SOURCE

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PREPARED:	May 6, 1987

BIBLIOGRAPHY

Jowise, P.P., Villnow, J.D., Gorelik, L.I., and Ryding, J.M., "Comparative Analysis of Soil Gas Sampling Techniques," Management of Uncontrolled Hazardous Waste Sites, Washington, D.C., December 1-3, 1986, p.193-199.

METHOD FM-14: SOIL GAS SAMPLING USING A PERFORATED TUBE

SUMMARY: Grab or composite soil gas samples obtained using pipe with perforated tip driven into ground. Analysis can be performed using portable GC, field laboratory and/or off-site laboratory.

METHOD DESCRIPTION: A non-reactive rod or pipe with a perforated tip is driven into the ground to the desired sampling depth. A pump is used to withdraw soil gas through Teflon tubing, from which grab samples can be taken using a syringe, or composite samples obtained by adding Tenax or other adsorbent material in the collection line along with a flow meter to measure air volume sampled. A water trap is used in some applications.

APPLICATION: Sampling of VOCs in the soil pore spaces in the unsaturated zone to assess extent of contamination.

LIMITATIONS: Aerobic degradation of hydrocarbons may occur in some areas at shallow sampling depths. Sample leakage from probe or syringe possible.

INSTRUMENTATION USED: Perforated pipe, Teflon tubing, gas-tight glass syringe, vacuum pump.

PERFORMANCE SPECIFICATION

DETECTION LIMIT: Not Known

SELECTIVITY: Volatile organics; affected by GC method used.

ACCURACY: Not Known

PRECISION/REPEATABILITY: Not Known

COMMENTS: Clay layers and horizons with < 5% air-filled porosity reduce effectiveness of soil gas sampling. Precision and ground water correlation must be demonstrated for each site. Determine applicability to each site by comparing to other sampling techniques.

USE

LOCATION USED: Tucson International Airport.

EPA SITE NUMBER (CERCLIS): CR811018010

MATRIX: Soil.

PREPARATION, MAINTENANCE AND CLEANUP: Flushing of sampling equipment with nitrogen gas is effective in preventing contamination. Inject carrier gas samples into GC to check for contamination. Draw air through soil gas probes and inject into GC to check for contamination. Teflon may be subject to carry-over from high liquid or gas-phase concentrations.

ANALYSIS TIME: NA

CAPITAL COST: Sampling probes ($100) each; syringes ($30-40), Sampling pump ($100-500). All are resusable. GC: $4,100 - $20,000

CALIBRATION: Pump should be calibrated if used for composite samples.

COMMENTS: One of several soil gas sampling methods that may be applicable under various circumstances.

PROTOCOL AVAILABLE: Yes.

SOURCE

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PREPARED: May 6, 1987

BIBLIOGRAPHY

Chapman, H., and Clay, P., Field Investigation Team (FIT) Screening Methods and Laboratories Complementary to Contract Laboratory Program, TDD-HQ-8507-01, October 17, 1986 (Draft)

Marrin, D.L., Thompson, G.M., Investigation of Volatile Contaminants in the Unsaturated Zone Above TCE Polluted Groundwater, EPA Project I.D. CR811018010, October 10, 1984.

METHOD FM-15: SOIL GAS SAMPLING USING TENAX TUBES

SUMMARY: Sampling (long-term and time-averaged) of soil gas for on-site analysis to determine volatile organic contaminant plume. Requires use of sampling probe and preconditioning of Tenax tubes. Analysis can be performed in a field laboratory or offsite laboratory.

METHOD DESCRIPTION: Stainless steel desorption tubes packed with Tenax are suspended inside a stoppered sampling probe and connected to a pump. Three liters of soil gas are drawn through each tube, concentrating the contaminant on the adsorbing material. In a field laboratory, contaminants are driven off thermally and analyzed using a GC.

APPLICATION: Soil sampling to aid in contaminant plume delineation, especially where very low concentrations expected.

LIMITATIONS: Sampling probe installation necessary. Tenax tubes must be preconditioned. Sampling requires lengthy pumping. Pre-packed Tenax tubes often contaminated; recommend packing own tubes, but requires laboratory and chemist.

INSTRUMENTATION USED: Tenax-filled desorption tubes; calibrated air pump; thermal desorber; gas-tight syringe.

PERFORMANCE SPECIFICATION

DETECTION LIMIT: Sensitivity improved over other sampling techniques by sample concentration; calculated 0.02 ug/l for TCE (10x below detection limit of GC used).

SELECTIVITY: Volatile organic contaminants; depends on GC method used for analysis.

ACCURACY: High correlation with other sampling techniques direct: injection-auger— 0.80; headspace sampler— 0.84; one-liter technique— 0.90; direct injection - stopper— 0.98. Also, high correlation with concentrations in water (0.80 & 0.99 in two sample groups). Percent recovery is unknown.

PRECISION/REPEATABILITY: RSD = 24% for 10 samples from a single location over a 4-week period. For three sets of triplicate samples from different locations, RSDs were 5%, 10%, and 44%, varying inversely with concentration.

COMMENTS: Variation in results is mainly due to the nature of soil gas sampling rather than the subsequent analysis unless contaminant concentrations are very low. Correlation with ground water contaminant concentrations may be misleading. Sensitivity depends on GC used; concentrating method overcomes some limitations of field GC. Clay horizons with <5% air-filled porosity reduce effectiveness of soil gas sampling. Should be compared with other sampling techniques to determine its applicability at a particular site.

USE

LOCATION USED: Puget Sound, WA, 1986.

EPA SITE NUMBER (CERCLIS): Not Available

MATRIX: Soil.

PREPARATION, MAINTENANCE AND CLEANUP: Preconditioning and assembly of Tenax tubes necessary (solvent extraction with methanol, then hexane; heating in N_2 atmosphere). Cleanup required for reuse.

ANALYSIS TIME: Drilling borehole— 15-30 minutes; installing sampling probe— 10 minutes; sample collection— 45 minutes; analysis— 25 minutes.

CAPITAL COST: Tenax tubes ($50-75); probes ($100); sampling pump ($100-500) Gas Chromatograph: $4,100 - $20,000

CALIBRATION: Sampling pump requires calibration, using a "dummy" Tenax tube and Buck Calibrator before and after sample collection.

COMMENTS: Allows long-term and time-averaged sampling. Comparatively easy sample collection. May not show long-term changes in soil gas concentration unless probe contents mixed to provide equilibrium along probe length before sampling. Provided more information about widespread, low-level contamination than Direct Injection - Auger method but no more detailed plume map. Allows more complete desorption than activated carbon and is easier to clean for reuse. Samples can be refrigerated for later analysis.

PROTOCOL AVAILABLE: No.

SOURCE

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BIBLIOGRAPHY

Jowise, P.P., Villnow, J.D., Gorelik, L.I., Ryding, J.M., "Comparative Analysis of Soil Gas Sampling Techniques," Management of Uncontrolled Hazardous Waste Sites, Washington, D.C. December 1-3, 1986, p. 193-199.

METHOD FM-16: SOIL GAS SAMPLING FOR DOWNHOLE PROFILING

SUMMARY: Soil gas samples are collected from borehole through Teflon tubing to determine vertical contamination gradient. Soil temperature increase due to boring may influence results. Analysis can be performed using portable GC or field laboratory.

METHOD DESCRIPTION: A Teflon collection hose with attached collection chamber lowered into a borehole, and a sample is withdrawn by pumping for analysis. Gas-tight syringe is used to take sample from a septum fitting on the pump. Grab or composite samples can be taken.

APPLICATIONS: Used to determine vertical contamination gradient, identify "hot spots," predict emission rates, and assess migration pathways as determined by soil type and stratigraphy.

LIMITATIONS: Uncontrolled variables include soil temperature influenced by heat generated by the auger and surface area of the soils.

INSTRUMENTATION USED: Custom fabricated downhole isolation flux chamber, Teflon line, gas-tight syringe.

PERFORMANCE SPECIFICATION

DETECTION LIMITS: Function of injection volume and detector sensitivity. 0.2 ug/l for TCE in one study.

SELECTIVITY: Volatile organics.

ACCURACY: Not Known

PRECISION/REPEATABILITY: Not Known

COMMENTS: Sensitivity depends on GC used. Clay layers and horizons with <5% air-filled porosity reduce effectiveness of soil gas sampling. Precision and ground water correlation must be demonstrated at each site. Compare with other sampling methods to determine applicability to the site.

USE

LOCATION USED: Not Available

EPA SITE NUMBER (CERCLIS): Not Available

MATRIX: Soil.

PREPARATION, MAINTENANCE, AND CLEANUP: Equipment decontamination required; purge syringes with nitrogen gas and check for contamination by GC analysis; check soil probes by drawing air through and analyzing. Teflon may be subject to carry-over from high liquid or gas-phase concentrations.

ANALYSIS TIME: 40 min/sample.

CAPITAL COST: Syringes ($30-40); sampling pump ($100-500). Both are reusable.

CALIBRATION: N/A

COMMENTS: One of several soil gas sampling methods; each has advantages under various circumstances.

PROTOCOL AVAILABLE: Yes.

SOURCE

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BIBLIOGRAPHY

Chapman, H., and Clay, P., "Field Investigation Team (FIT) Screening Methods & Mobile Laboratories Complementary to Contract Laboratory Program (CLP)," TDD-HQ-8507-01, October 17, 1986 (Draft).

METHOD FM-17: SOIL GAS SAMPLING USING DIRECT INJECTION - AUGER (DIA)

SUMMARY: Rapid sampling technique for soil gas analysis to aid in ground water plume determination. Repeated sampling not possible once auger is pulled. Analysis can be performed using a portable GC and/or field laboratory.

METHOD DESCRIPTION: A 12'-17'-foot borehole is drilled using a hollow-stem auger (8" O.D.). When desired sampling depth is reached, a 500 ml gas-tight, side port syringe is lowered and filled. Syringe contents are injected directly into a GC.

APPLICATION: Delineation of extent of contamination; rapid soil gas sampling.

LIMITATIONS: No repeated sampling. Lack of control of sampling environment.

INSTRUMENTATION USED: Auger for drilling and 500 ml gas-tight, side port syringe for sampling; GC for analysis.

PERFORMANCE SPECIFICATION

DETECTION LIMIT: Heat generated by drilling raises sampled concentration of contaminants; however, fewer DIA samples found contaminant, possibly indicating less sensitivity than other soil sampling methods. 0.2 ug/l for TCE in one study, based on capability of GC used for analysis.

SELECTIVITY: Volatile organics; depends on GC method used.

ACCURACY: Results correlated well with results from other sampling methods at same locations: Correlation coefficients were 0.68 for one-liter method; 0.72— headspace method; 0.78— direct injection-stopper; 0.80— Tenax.

PRECISION/REPEATABILITY: Not Known.

COMMENTS: May not accurately reflect ground water conditions due to quick nature of sampling. GC used will affect detection limits. Clay layers and horizons with <5% air-filled porosity reduce effectiveness of soil gas sampling. Compare method to other sampling techniques to determine its applicability to a particular site.

USE

LOCATION USED: Puget Sound, WA, 1986.

EPA SITE NUMBER (CERCLIS): Not Available

MATRIX: Soil

PREPARATION, MAINTENANCE AND CLEANUP: Determine appropriate drilling depth by use of test holes (need to drill below surface soils to bypass competing adsorption sinks) or by combined experience and assessment of site factors. Decontamination of auger and syringes required.

ANALYSIS TIME: 15-30 minutes to drill borehole; 1 minute for sample collection.

CAPITAL COSTS: Syringes: ($30-40); GC: $4,100 - $20,000.

CALIBRATION: Purge syringes with nitrogen and check for contamination by injecting carrier gas samples into GC.

COMMENTS: Sample collection relatively easy. Minimal sample preparation required before analysis.

PROTOCOL AVAILABLE: Yes.

SOURCE

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PREPARED: May 6, 1987

BIBLIOGRAPHY

Jowise, P.P., Villnow, J.D., Gorelik, L.I., and Ryding, J.M., "Comparative Analysis of Soil Gas Sampling Techniques," Management of Uncontrolled Hazardous Waste Sites, Washington, D.C., December 1-3, 1986., p. 193-199.

METHOD FM-18: PCB ANALYSIS USING A GAS CHROMATOGRAPH IN AN ON-SITE LABORATORY— HEXANE/METHANOL/WATER EXTRACTION

SUMMARY: Rapid determination of major Aroclors to 200 ug/kg in soil. Accurate to 100,000 ug/kg, then underestimates by 60%. Requires field lab. Appropriate extraction solvent should be determined by laboratory testing prior to field use.

METHOD DESCRIPTION: Requires a field laboratory with a GC and linearized electron capture detector for PCB analysis of soil samples. Identification is done by comparing peak retention time with external standards. Quantitation is determined by comparing peak heights and volumes of the standard and sample. Sample preparation consists of mixing 0.8 gram of soil with with a 1:4:5 ratio of distilled water/methanol/hexane. An optional step is to dry and grind sample before extraction. Agitate sample and let sit, allowing hexane layer to separate. Transfer hexane layer to a test tube containing sulfuric acid and mix. This step is optional, as it is used to eliminate matrix interferences. Withdraw sample from hexane layer for GC analysis.

APPLICATION: Simple and rapid determination of polychlorinated biphenyls. Method most appropriate for Aroclors 1242, 1248, 1254 and 1260, but good for 1016, 1221, and 1232.

LIMITATIONS: Above 100,000 ug/kg, concentrations are underestimated by 60%. Results are approximations.

INSTRUMENTATION USED: Analytical Instrument Development Corp. (AID) Model 511-06 GC equipped with ECD and a 4-foot SE-30 stainless column.

PERFORMANCE SPECIFICATION

DETECTION LIMIT: 200 ug/kg

SELECTIVITY: Compounds give characteristic multiple peaks, good selectivity.

ACCURACY: Recovery of Aroclor 1242 spike = 80%-105%. Based on results of 300 samples, accuracy is equivalent to CLP below 100,000 ug/kg; determination above 100,000 ug/kg are biased low (about 40% of the CLP determined value).

PRECISION/REPEATIBILITY: Relative Standard Deviation (RSD)of 4 samples = 10%-12%.

COMMENTS:

USE

LOCATION USED: Extensively used; sites include Washburn, ME; Norwood, MA.

EPA SITE NUMBER (CERCLIS): Not Available

MATRIX: Soil.

PREPARATION, MAINTENANCE AND CLEANUP: Care should be taken not to contaminate column. Before run, column should be at thermal equilibrium.

ANALYSIS TIME: 5-10 samples/hr.

CAPITAL COST: $7,245.00

CALIBRATION: Calibration determined by peak heights and retention times of PCB standards. Standards and blanks should be run every tenth sample.

COMMENTS: This method has been used often in Region I.

PROTOCOL AVAILABLE: Yes.

SOURCE

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PREPARED: 4/9/87

BIBLIOGRAPHY

Spittler, T.M., "Field Measurement of PCBs in Soil and Sediment Using a Portable Gas Chromatograph." Proceedings of 4th National Conference on Management of Hazardous Waste Sites. Washington, DC, October 31-November 2, 1983.

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METHOD FM-19: PCB Analysis Using a Gas Chromatograph in an On-site Laboratory - Hexane Extraction.

SUMMARY: Rapid determination of major Aroclors to 25 ug/l in water and 2,500 ug/kg in soil. Requires field laboratory. Appropriate extraction solvent should be determined by laboratory testing prior to field use.

METHOD DESCRIPTION: Requires field laboratory with GC and linearized ECD for PCB analysis of water and soil samples. Identification is done by comparing peak retention times with external standards. Quantitation is determined by comparing peak heights and volumes of the standard and sample. Sample preparation for water consists of adding 1.5 ml hexane to 15 ml of water, mixing and separating hexane layer. Sample preparation for soil consists of mixing 2 grams of soil with 2 grams sodium sulfate. Then 10 ml of hexane is added to sample, mixed with an ultrasonic probe and hexane layer separated. The hexane layer is ready for GC analysis.

APPLICATION: Simple and rapid determinations of polychlorinated biphenyls (PCBs). Also determines pesticides. (Testing based on Aroclor 1260).

LIMITATIONS: Results are semi-quantitative. Has not been used in the field.

INSTRUMENTATION USED: Hewlett-Packard 5880 with ECD.

PERFORMANCE SPECIFICATION

DETECTION LIMITS: 25 ug/l in water and 2,500 ug/kg in soil.

SELECTIVITY: Compounds give characteristic, multiple peaks; good selectivity.

ACCURACY: Not available.

PRECISION/REPEATABILITY: Not available.

COMMENTS: This method was developed by Region IV to provide semiquantitative data. Until recently, not used as a quantitative method.

USE

LOCATION USED: Field Mobile Laboratory in Florida

EPA SITE NUMBER (CERCLIS): N/A

MATRIX: Soil and Water

PREPARATION, MAINTENANCE AND CLEANUP: Precautions should be taken not to contaminate column. Occasional cleaning and reconditioning of column and detector required.

ANALYSIS TIME: 20-25 min/sample

CAPITAL COST: $20,000.00

CALIBRATION: Calibration determined by peak area and retention time of PCB standard.

COMMENTS: A much less expensive GC/ECD could be used.

PROTOCOL AVAILABLE: Yes.

SOURCE

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PREPARED: April 15, 1987

BIBLIOGRAPHY

"Screening Method For Extractable Organic Compounds," EPA Region IV, Mobile Laboratory Protocol, January 1987.

METHOD FM-20: PCB ANALYSIS USING A GAS CHROMATOGRAPH IN AN ON-SITE LABORATORY— HEXANE/METHANOL

SUMMARY: Rapid determination of major Aroclors to 200 ug/kg in soil and 200 ug/l in water. Requires field laboratory. Appropriate extraction solvent should be determined by laboratory testing prior to field use.

METHOD DESCRIPTION: Requires a field laboratory with GC and linearized electron capture detector for PCB analysis of soil samples. Identification is done by comparing peak retention times with external standards. Quantitation is determined by comparing peak heights and volumes of the standard and sample. Sample preparation for water consists of adding 1 ml hexane to 100 ml of water, mixing sample, and separating hexane layer. This step is repeated once. Add 1 ml sulfuric acid to hexane extract and mix. Sample is ready for GC analysis. Sample preparation for soil consists of mixing 1 to 2 grams soil, 2 ml methanol, and 10 ml hexane. Separate hexane layer, add 1 ml sulfuric acid to extract, and mix. Hexane extract is ready for GC analysis.

APPLICATION: Simple and rapid determination of polychlorinated biphenyls. Method most appropriate for Aroclor 1232, 1242, 1248, 1254, 1260.

LIMITATIONS: Results are approximations.

INSTRUMENTATION USED: Analytical Instrument Development Corp. (AID) Model 511-06 with ECD and 4-ft SE-30 column; or Shimadzu Mini-2 with ECD and 4-ft OV-1 column.

PERFORMANCE SPECIFICATION

DETECTION LIMIT: 200 ug/l in water
100 ug/kg in soil

SELECTIVITY: Compounds give characteristic multiple peaks, good selectivity.

ACCURACY: Average Percent Recovery = 104% (Range 65%-193%).

PRECISION/REPEATABILITY: Average Relative Standard Deviation = 14%; range 5.0%-42%.

COMMENTS: The percent recovery decreases as PCB concentration increases.

USE

LOCATION USED: Beaver Creek, Oregon 1985

EPA SITE NUMBER (CERCLIS): Not Available

MATRIX: Soil and Water

PREPARATION, MAINTENANCE AND CLEANUP: Care should be taken not to contaminate column. Column should be at thermal equilibrium before running.

ANALYSIS TIME: 5-10 samples/hr

CAPITAL COST: AID-511 $7,245.00
Shimadzu
Mini-2
$4,520.00

CALIBRATION: Calibration determined by peak heights and retention times of PCB standards. Standards and blanks should be run every tenth sample.

COMMENTS: Used regularly in Region X.

PROTOCOL AVAILABLE: Yes.

SOURCE

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PREPARED	April 9, 1987

BIBLIOGRAPHY

Chapman, H. and Clay, P., "Field Investigation Team (FIT) Screening Methods and Mobile Laboratories Complementary to Contract Laboratory Program (CLP)," TDD-HQ-8507-01, October 17, 1986. (Draft).

METHOD FM-21: PCB ANALYSIS USING A GAS CHROMATOGRAPH IN AN ON-SITE LABORATORY— HEXANE/ACETONE EXTRACTION

SUMMARY: Rapid determination of major Aroclors down to 2,000 ug/kg in soil. Requires field laboratory. Method was not tested for all PCBs. Appropriate extraction solvent should be determined by laboratory testing prior to field use.

METHOD DESCRIPTION: Requires field laboratory with a gas chromatograph (GC) and linearized electron capture detector (ECD) for PCB analysis of soil samples. Identification is done by comparing peak retention times with external standards. Quantitation is determined by comparing the peak heights and volumes of the standard and sample. Sample preparation consists of mixing 10-15 grams of soil with a UV grade 1:1 hexane/acetone solution, followed by extraction. Florisil "SepPak" used to adsorb interferences from solution. Extract from SepPak then screened by GC analysis.

APPLICATION: Simple and rapid determination of polychlorinated biphenyls; Aroclor 1232, 1242, 1248, 1254, and 1260.

LIMITATIONS: Method was not tested for all PCBs.

INSTRUMENTATION USED: Hewlett Packard (HP) Model 5840-A Gas Chromatograph with one glass column; or HP Model 5880-A Gas Chromatograph with one glass column; with AID 511 or Shimadzu Mini-2 with Electron Capture Detector

PERFORMANCE SPECIFICATION

DETECTION LIMIT: 2,000 ug/kg

SELECTIVITY: Peaks are separate; good selectivity.

ACCURACY: Approximately ±5% of true value. Mean recovery of soil spike = 97%-98% (Tested for eleven soil spikes).

PRECISION/REPEATABILITY: One sample run in triplicate gave standard deviation of 0.231. Two duplicate samples differed by 2-18%. Relative standard deviation (RSD) of soil spike was ± 14% (eleven samples).

COMMENTS: Samples below the detection limit can be concentrated and re-run.

USE

LOCATION USED: G.E Moreau, Schenectady N.Y.; Modified at Wide Beach, N.Y.

EPA SITE NUMBER (CERCLIS): NYD980528335

MATRIX: Soil

PREPARATION, MAINTENANCE AND CLEANUP: After a week of continuous use, columns and detectors have to be cleaned and reconditioned, requiring one and a half days of down time.

ANALYSIS TIME: 30 minutes per sample.

CAPITAL COSTS: $18,000.00

CALIBRATION: Calibration determined by the peak heights and retention times of PCB standards. Standards and method blanks are run after every tenth sample.

COMMENTS: This method has had very limited use.

PROTOCOL AVAILABLE: No.

SOURCE

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PREPARED: April 17, 1987

BIBLIOGRAPHY

A Rapid Procedure for the Determination of Polychlorinated Biphenyls in Soils. Draft. U.S. EPA Region II.

METHOD FM-22: PESTICIDE ANALYSIS USING A GC WITH ECD— HEXANE/METHANOL EXTRACTION

SUMMARY: Rapid determination of most pesticides to 100 ug/l in water and to 20 ug/kg in soil. Low recovery of some pesticides. Field laboratory required.

METHOD DESCRIPTION: Requires field laboratory with GC and linearized ECD for pesticide analysis in soil and water. Identification and quantitation is determined by comparing peak retention times and peak areas, respectively, of the standard and sample. Sample preparation for water consists of adding 1 ml hexane to 100 ml of water, mixing, and separating hexane layer. This step is repeated once. Sample preparation for soil consists of mixing 1 to 2 grams of soil, 2 ml methanol and 10 ml hexane, and separating the hexane layer. 1 ml sulfuric acid is added to the extract and mixed. If pesticide is sensitive to acidification, this step is omitted. Hexane extract is ready for GC analysis.

APPLICATION: Simple and rapid determination of most pesticides except for endrin ketone and methoxychlor. Also determines PCBs.

LIMITATIONS: Poor spike recoveries for some pesticides. Method requires testing before use. Results are semi-quantitative.

INSTRUMENTATION USED: Analytical Instrument Development Corp. (AID) model 511-06 with ECD and 4 ft SE-30 column; or Shimadzu Mini-2 with ECD and 4 ft OV-1 column.

PERFORMANCE SPECIFICATION

DETECTION LIMITS: 100 ug/l in water and 20 ug/kg in soil.

SELECTIVITY: Compounds give separate peaks, good selectivity.

ACCURACY: 3 water matrix spikes of 13 compounds: recovery = 8%-107%.
3 soil matrix spikes of 13 compounds: recovery = 26%-200%.

PRECISION/REPEATABILITY: Relative Standard Deviation (RSD) of 13 compounds in water, in triplicate, RSD = 1.5%-34.2%; 13 compounds in soil, in triplicate, RSD = 7.6%-54.9%.

COMMENTS: Recoveries are given before and after acidification.

USE

LOCATION USED: Beaver Creek, Oregon 1985.

EPA SITE NUMBER (CERCLIS): ORD095016887

MATRIX: Soil and Water

PREPARATION, MAINTENANCE AND CLEANUP: Column should be at thermal equilibrium before running. Column and detector require occasional cleaning and reconditioning.

ANALYSIS TIME: 30-45 min/sample

CAPITAL COST: AID 511 with ECD $7,245.00 Shimadzu Mini-2E with ECD $4,520.00

CALIBRATION: Calibration determined by peak retention times and areas of PCB standards. Standards and method blank should be run every tenth sample.

COMMENTS: Used fairly regularly in Region X.

PROTOCOL AVAILABLE: Yes.

SOURCE

TECHNICAL CONTACT: Hunt Chapman
AFFILIATION: Ecology and Environment, Inc.
TELEPHONE: (703) 522-6065
PREPARED: April 15, 1987

BIBLIOGRAPHY

Chapman, M. and Clay, P. "Field Investigation Team (FIT) Screening Methods and Mobile Laboratories Complementary to Contract Laboratory Program (CLP)," TDD-HQ-8507-01, October 17, 1986 (Draft).

METHOD FM-23: PESTICIDE ANALYSIS USING ISOTHERMAL GC WITH ECD— HEXANE EXTRACTION

SUMMARY: Rapid determination of pesticides to 5 ug/l in water and 500 ug/kg in soil. Requires field lab. Many other compounds detected with pesticides on GC.

METHOD DESCRIPTION: Requires field laboratory with GC and linearized ECD for pesticide analysis in soil and water. Identification and quantitation is determined by comparing peak retention times and peak areas of the standard and sample. Sample preparation for water consists of mixing 15 ml of water with 1.5 ml hexane and separating hexane layer. Sample preparation for soil consists of mixing 2 grams soil with 2 grams sodium sulfate. To this, 10 ml hexane is added, mixed with an ultrasonic probe, and hexane layer separated. The hexane layer is ready for analysis.

APPLICATION: Simple and rapid determination of pesticides. Also detects PCBs.

LIMITATIONS: Results are semi-quantitative. Has not been used in the field. Detects phosphorus, nitrogen, sulfur, and oxygen compounds along with pesticides.

INTRUMENTATION USED: Hewlett-Packard Model 5880 with ECD.

PERFORMANCE SPECIFICATION

DETECTION LIMIT: 5 ug/l in water and 500 ug/kg in soil.

SELECTIVITY: Compounds give separate peaks, many compounds detected; usually good selectivity.

ACCURACY: Not available.

PRECISION/REPEATABILITY: Not available.

COMMENTS: This method was developed by Region IV to provide tentative identification of compounds and semiquantitative data. Until recently not used as a quantitative method.

USE

LOCATION USED: Field Mobile Laboratory in Florida

EPA SITE NUMBER (CERCLIS): Not Applicable.

MATRIX: Soil and water

PREPARATION, MAINTENANCE AND CLEANUP: Precautions should be taken not to contaminate column. Occasional cleaning and reconditioning of column and detector required.

ANALYSIS TIME: 20-25 min/sample.

CAPITAL COST: $20,000.00

CALIBRATION: Calibration determined by peak areas and retention times of pesticide standards.

COMMENTS: A much less expensive GC/ECD could be used.

PROTOCOL AVAILABLE: Yes.

SOURCE:

TECHNICAL CONTACT: William Loy

AFFILIATION: EPA Region IV ESD

TELEPHONE: (404) 546-3386

PREPARED: April 15, 1987

BIBLIOGRAPHY

"Screening Method For Extractable Organic Compounds", EPA Region IV Mobile Laboratory Protocol, January 1987.

METHOD FM-24: PHENOL DETERMINATION BY LIQUID-LIQUID EXTRACTION AND GC ANALYSIS

SUMMARY: Detection of phenols in water to 100 ug/l and soil to 25 ug/kg. Requires field laboratory and chemist.

METHOD DESCRIPTION: Uses liquid-liquid extraction with GC analysis to determine phenols in soil and in water. The procedure for soil analysis entails taking 2-3 grams of soil and extracting with methanol. The procedure for water analysis consists of taking 20 ml of water, adjusting the pH to 2, and extracting with methanol. Extract for both soil and water is reacted with pentafluorobenzyl bromide and potassium carbonate in presence of hexacyclooctadecane catalyst to form pentafluorobenzyl (PFB) phenol derivative. PFB derivative is exchanged into hexane using liquid-liquid extraction. An aliquot of hexane solution is injected into GC for analysis. Identification and quantitation by comparison of retention time and peak height and volume to standard (response factor determination required). Method is a modification of EPA method 604.

APPLICATION: Determination of phenol concentrations in soil and water; 2,4-dimethyl phenol, phenol, 2-chlorophenol, 2-nitrophenol, 2,4-dichlorophenol, 4-chloro-3-methylphenol, 2,4,6-trichlorophenol and pentachlorophenol.

LIMITATIONS: Liquid-Liquid extraction procedure is a difficult process and can best be performed by a trained chemist.

INSTRUMENTATION USED: Shimadzu GC Mini-2 Gas Chromatograph with ECD and Shimadzu Chromatopac C-RSA Data Processor.

PERFORMANCE SPECIFICATION:

DETECTION LIMITS: Detection limit for pentachlorophenol in water = 100 ug/l and in soil = 25 ug/kg

SELECTIVITY: Separate peaks, easily identifiable. Other compounds may appear on chromatograph.

ACCURACY: Spike recoveries in soil (6 replicates of 8 compounds) = 102%-127%. Spike recoveries in water (6 replicates of 8 compounds) = 1.0%-88%.

PRECISION/REPEATABILITY: Relative Standard Deviation (RSD) for 6 replicates of 8 compounds in soil = 15.3%-44.4%. RSD for 6 replicates of 8 compounds in water = 19.6% to 56.2%.

COMMENTS: Recoveries for water samples were acceptable for pentachlorophenol (88%, CLP limits = 9-103%) and phenol (25.8%, CLP limits = 12-89%).

USE

LOCATION USED: Not Available

EPA SITE NUMBER (CERCLIS): Not Applicable

MATRIX: Water and Soil

PREPARATION, MAINTENANCE AND CLEANUP: Column cleaning and reconditioning required occasionally. Column can be saturated easily by concentrated samples.

ANALYSIS TIME: 20-40 min/sample, depending on column length

CAPITAL COST: $4520 Shimadzu GC with ECD

CALIBRATION: Standards necessary to identify and quantify compounds. Calibration is done using mixed standard solutions of known concentrations. A minimum of three standard solutions should be used for calibration.

COMMENTS: Interpretation of results requires a trained chemist. Used occasionally in Region X.

PROTOCOL AVAILABLE: Yes

SOURCE

TECHNICAL CONTACT: John Ryding
AFFILIATION: C.C. Johnson & Malhotra
TELEPHONE: (303) 433-6966
PREPARED: April 22, 1987

BIBLIOGRAPHY

McGinnis, Roger, "Screening Method for Acid Extractables (Phenols) in Soil and Water," EPA Document No. TDD-R10-8601-04, September, 1986.

METHOD FM-25: PAH ANALYSIS USING GC (FID) WITH HEATED COLUMN

SUMMARY: Detects PAHs in water and in soil to ppm range. GC may give overlapping peaks, hindering identification. Has trouble determining naphthalene.

METHOD DESCRIPTION: Requires a field laboratory with a gas chromatograph able to heat column above ambient temperatures for polycyclic aromatic hydrocarbon (PAH) determination. Identification and quantitation is done by comparing peak retention times and peak areas between standards and samples. Procedure for water analysis consists of extracting contaminants from 100 ml water sample into 1 ml methylene chloride by vortex mixing. Procedure for soil analysis consists of mixing 2-3 grams of soil with 6 ml methylene chloride and separating methylene chloride. Extraction step for soil and water is repeated, and extracts are combined. The methylene chloride is passed through a silica gel column to remove potential interferences before GC analysis. Method is a modification of EPA method 610.

APPLICATION: Rapid identification and quantitation of polycyclic aromatic hydrocarbons.

LIMITATIONS: Possible co-eluting peaks (e.g., phenanthrene and anthracene). Method gives poor spike recovery for naphthalene. Does not detect many semi-volatiles.

INSTRUMENTATION USED: Shimadzu Mini-2F or Shimadzu Mini-3 GC (FID) with 6 foot glass quantitation and confirmation column.

PERFORMANCE SPECIFICATION

DETECTION LIMITS: 500-1000 ug/l in water and 50-500 ug/kg in soil for PAHs.

SELECTIVITY: Peaks usually separate and give good selectivity. Occasional co-eluting peaks.

ACCURACY: Spike recovery for 12 different compounds in soil: 67%-129% (naphthalene excluded, 14% recovery).

PRECISION/REPEATABILITY: Relative Standard Deviation (RSD) for 12 different compounds in soil: 7.8%-55.7%.

COMMENTS: None

USE

LOCATION USED: J.H. Baxter, Washington, 1986

EPA SITE NUMBER (CERCLIS): WAD009265521
WAD053823019

MATRIX: Soil and Water

PREPARATION, MAINTENANCE AND CLEANUP: Columns and detectors require occasional cleaning and reconditioning.

ANALYSIS TIME: 60 min/sample

CAPITAL COST: Shimadzu Mini-2F— $4,100.00; Mini-3— $5,200.00

CALIBRATION: Retention times and peak areas of standards must be obtained for the compounds of interest. Standards are used to generate a three-point calibration curve.

COMMENTS: Used fairly regularly in Region X.

PROTOCOL AVAILABLE: Yes.

SOURCE

TECHNICAL CONTACT: Hunt Chapman
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TELEPHONE: (703) 522-6065
PREPARED: April 14, 1987

BIBLIOGRAPHY

Chapman, H. and Clay, P., "Field Investigation Team (FIT) Screening Methods and Mobile Laboratories Complementary to Contract Laboratory Program (CLP)," EPA Document Number TDD-HQ-8507-01, October 17, 1986. (Draft).

METHOD FM-26: Total PNA Analysis using an Ultraviolet Fluorescence Spectrophotometer

SUMMARY: Rapid, semi-quantitative determination of total polynuclear aromatics (PNAs) in soil and water to 10.0 ug/l in water and 1,000 ug/kg in soil; field laboratory required.

METHOD DESCRIPTION: Requires a field laboratory with UV fluorescence spectrophotometer. The compounds' concentrations will determine the amount of a particular wavelength of light absorbed. Method consists of measuring a set amount of soil or water, a one-step extraction followed by UV fluorescence spectrophotometric analysis. Analysis occurs at ambient temperature; constant temperature preferable. Sodium sulfate is added to soil samples and is used as an absorbent for water extract samples to clean up the sample. Acetonitrile is used for soil extraction and hexane for water extraction. Quantitation is done by a seven-point calibration curve.

APPLICATION: Simple and rapid determination of total PNAs.

LIMITATIONS: Does not identify individual PNA compounds. Must be validated for each site due to potential matrix interferences.

INSTRUMENTATION USED: Perkin-Elmer Model LS-5 Fluorescence spectrometer with chart recorder.

PERFORMANCE SPECIFICATION

DETECTION LIMIT: 10.0 ug/l in water, 1,000 ug/kg in soil.

SELECTIVITY: Dependent upon matrix interference. Where used, only PNAs were detected.

ACCURACY: Spike recoveries for napthalene/acenapthene for soil (10 samples) and water (6 samples) were 63%-100% and 81%-101% respectively. Spike recoveries for phenanthrene for soil (10 samples) and water (6 samples) were 66%-130% and 93%-111%, respectively.

PRECISION/REPEATABILITY: Relative Standard Deviation (RSD) for phenanthrene for water (6 samples) and soil (10 samples) were 0.6%-11% and 0.6%-13%, respectively. RSD for napthalene/acenaphthene for water (6 samples) and soil (10 samples) were 1.2%-10% and 0%-12.7%, respectively.

COMMENTS: Specifications shown based on data from two sites. Calibration curves extrapolated to non-linear response regions will give high concentrations (>100% recoveries of spikes). Results were within an order of magnitude of GC/MS results, which is considered acceptable for screening.

USE

LOCATION USED: Southern Maryland Wood Treating Plant. Summer 1986.

EPA SITE NUMBER (CERCLIS): MDD980704852

MATRIX: Soil and water.

PREPARATION, MAINTENANCE AND CLEANUP: UV Fluorescence cells should be cleaned occasionally.

ANALYSIS TIME: 20-30 samples/day

CAPITAL COST: $16,300.00

CALIBRATION: Standards were made of seven commonly occurring PNAs. Calibration curve was from 0.01 g/l to 0.1 g/l and 0.1 g/l to 1.0 g/l, with four points for each curve.

COMMENTS: Can be operated by trained technician.

PROTOCOL AVAILABLE: No.

SOURCE

TECHNICAL CONTACT: Stacie Popp

AFFILIATION: Roy F. Weston, Inc.

TELEPHONE: (215) 692-3030

PREPARED: April 10, 1987

BIBLIOGRAPHY

Motwani, J.N., Popp, S.A., Johnson, G.M. and Mindock, R.A., "Field Screening Techniques Developed Under the Superfund Program," Management of Uncontrolled Hazardous Waste Sites, Washington, D.C., December 1-3, 1986.

METHOD FM-D1: TRACE ATMOSPHERIC GAS ANALYZER (TAGA) (METHOD UNDER DEVELOPMENT)

SUMMARY: Quick characterization and monitoring of many organic compounds, using MS/MS. Capable of analyzing direct air samples for most organics at the ppb level, and performing rapid GS/MS/MS analyses of soils. Capable of real-time screening of ambient air for target compounds (1/2 order of magnitude accuracy), but the quantitative data reduction (increase of accuracy) to include the calculation of error bars requires addition time. Some interferences possible with hydrocarbons and chlorinated solvents; lacks isomer specificity; requires frequent recalibration; standard QA/QC procedures not compatible. Analyses are performed in specially build mobile laboratory.

METHOD DESCRIPTION: Triple quadrupole MS/MS analyzes polar and non-polar compounds in ambient air using direct sampling. The MS/MS uses the ambient air as its chemical ionization reagent gas. Chemical ionization of target compounds achieved by charge transfer, hydride abstraction, halide abstraction, hydride-halide abstraction, protonation, and/or adduct formation. Ionization is determined by the compound class being analyzed and the instrument source being used. In tandem mode, preselected ions are fragmented and analysed to produce characteristic mass spectra; specific fragment ions can be sought. In single mass analyzer mode, scans all ions produced. For air analyses, chromatographic separation is not compatible with the existing software. Some type of parallel GC sampling is recommended for compound confirmation. Ion signals obtained in the tandem mode can be used to directly calculate air concentration.

APPLICATION: For air and soil gas samples - instant (real-time) screening and monitoring of concentration changes over time (e.g., plume tracking and characterization) for polar and nonpolar compounds including amines, nitrogen and sulfur containing heterocyclic compounds, oxygenated hydrocarbons, halogenated hydrocarbons, aromatics, C5 and larger hydrocarbons, and sulfur containing hydrocarbons. Also capable of quantitation but more analytical control and data reduction time needed. For soil analyses - capable of "dilute and shoot" GC/MS/MS analyses for any GC compatible compound present in mg/kg concentrations (e.g., PCBs and chlorinated pesticides) and "rapid cleanup and shoot" GC/MS/MS analyzes for any compound present in ug/kg concentrations (e.g., TCDD).

LIMITATIONS: Lacks isomer specificity for air analyses. Hydrocarbons and chlorinated solvents can yield ions of equal mass and similar structures, resulting in cross interferences. Dirty samples can yield spectra with fragments from more than one parent structure. Not as stable as normal GC/MS (2- to 3-fold drift in sensitivity over a day is possible). The sampling system is not totally compatible with classical QA/QC procedures (direct sampling of audit cyclinders and replicate analyses) but TAGA specific QA/QC procedures are currently being developed and refined by the ERT and NYDEC. Difference in ionization and the use of an initial mass filtering will result in MS/MS spectra being different than the MS spectra contained in standard EPA/NIH standard spectral libraries.

The use of the sample matrix as the chemical ionization reagent gas can result in potential matrix affects. The current instrument software does not support QA/QC criteria being applied to quantitative results obtained during air analyses. Quantitative software utilizing QA/QC criteria is currently being developed by the ERT and by NYDEC. Similar software development is also being proposed by Battelle and by Sciex, the instrument manufacturer. For all analyses - because of its cryogenic vacuum pump the instrument can only be used for a maximum of sixteen hours a day. However, typical analysis days rarely exceed fourteen hours.

INSTRUMENTATION USED: TAGA 6000E Triple Quadrupole Mass Spectrometer/Mass Spectrometer; three quadrupoles are RF-coupled; sources in use are Atmospheric Pressure Chemical Ionization (APCI) and Low Pressure Chemical Ionization (LPCI).

PERFORMANCE SPECIFICATION

DETECTION LIMIT: 1-10 ug/l for most non-polar organics in air; 0.02-1.7 ug/kg for dioxins in soil (0.02 ug/kg for 2,3,7,8-TCDD); 25 ug/l for phenols and other such polar compounds in air; 1 mg/kg for PCB and chlorinated pesticides in soil (without cleanup).

SELECTIVITY: Compounds may be identified by comparing spectral peaks; but lacks isomer specificity in air analyses. Also, during air analyses certain non-isomeric compounds will form isomers upon ionization (e.g., isopropanol and acetone, 1,2-dichloroethane and vinyl chloride, and methylene chloride and chloroform).

ACCURACY: Variation of less than 20% between spiked PCB and TCDD samples in different soil types.

PRECISION/REPEATABILITY: Standard deviation of 0.68 with a mean of 5.5 ug/l dioxin (repeated analyses of well homogenized soil). Relative standard deviation of 22% for nine spiked soil samples for PCBs.

COMMENTS: Flash chromatography can be used as a gross cleanup step before MS/MS when interferences are likely, e.g., isobaric compounds with overlapping collision-induced dissociation spectra or compounds that react preferentially with the reagent gas. Accurate quantification difficult in air samples if >10,000 ug/l methane is present. Because of the possibilities of self-chemical ionization and source chemistry saturation, quantitation in the mg/l range is suspect. The quantitation of many compounds in air is very sensitive to changes in the absolute humidity of the air sampled, especially with the APCI source. Quantitation of polar compounds by the APCI source is very sensitive to the presence of ammonia and mg/l levels of amines. LPCI is required source for most environmental sampling.

USE

LOCATION USED: Oceanside, NJ; Utica, NY; and numerous other sites.

EPA SITE NUMBER (CERCLIS): Not Available

MATRIX: Air, (ambient and indoor) soil, water, soil gas and incinerator wastes.

PREPARATION, MAINTENANCE AND CLEANUP: Soil sample preparation for dioxin analysis typically consists of a single-step extraction, a rapid dual mini-column cleanup (8 samples every 3-4 hours), and flash gas chromatography (temperture program at 25 C /min.). Soil preparation for PCBs and chlorinated pesticides at mg/kg concentrations consists of single-step extraction, extract dilution, and flash chromatography ("dilute and shoot"). Water analyses consist of extraction and flash chromatography of the extract. Air analyses typically consist of sampling the air directly from a 1.5-2 liter/second sample stream.

ANALYSIS TIME: Dioxin— 15-20 minutes for non-isomer specific analyses; 30-45 minutes for TCDD; complete screening of chemical classes in air— 30 minutes; complete set of air analyses for a set of target compounds— 1-10 seconds depending on the number of compounds.

CAPITAL COSTS: $500,000 for MS/MS; $800,000 in fully outfitted van.

CALIBRATION: For GC/MS/MS analyses— uses conventional GC calibration methods. For air analyses two different calibration techniques are used: 1) A motorized syringe drive expels headspace vapor from syringe containing solid or liquid. Vapor from syringe is mixed with air at 2 liters/minute in the temperature-controlled (100oC) mixing tee. Relative concentration in the stream is calculated based on vapor pressure and flow rates. Overall calibration accuracy is + 30%. 2) Certified gas standard cylinders (25 - 50 ppm(v)) can also be serially diluted directly into the sampled air stream to generate calibration concentrations in the range of 5 - 100 ppb(v). This method has an overall calibration accuracy of + 10% but is limited only for compounds which can be maintained at stable concentrations in gas cylinders.

For air analyses the instrument should be calibrated for each target compound at least twice daily. In addition, for many target compounds (e.g., tetrachloroethylene, chlorinated aromatics, and oxygenated solvents) the calibrations should be periodically checked throughout the day.

COMMENTS: Requires high level of expertise and experience on the part of the analyst. Calibration is simple, and method is relatively fast, cost-effective, and applicable to many compounds. Calibration accuracy depends on accuracy of literature value of vapor pressure vs. temperature and on setting of flow rates. Use of ambient air as reagent gas means that as air changes, calibrations change.

PROTOCOL AVAILABLE: Available upon request for TCDD. Air protocols currently under revision to incorporate ERT developed software and ERT/NYDEC developed QA/QC procedures. Onsite consultation available from ERT.

SOURCE

TECHNICAL CONTACT:	Tom Pritchett/Dr. S.H. MO
AFFILIATION:	EPA ERT/NYDEC
TELEPHONE:	(201) 321-6738/(518)457-7454
PREPARED:	April 29, 1987

BIBLIOGRAPHY

Engels, J.W., Kerfoot, H.B., and Arnold, D.F., Survey of Mobile Laboratory Capabilities and Configurations, EPA 600/X-84-170, U.S. Environmental Protection Agency, July 1984.

Ben-Hur, D., Smith, J.S., and Urban, M.J., Application of Mobile MS/MS to Hazardous Waste Site Investigation, 5th National Conference on Management of Uncontrolled Hazardous Waste Sites, November 7-9, 1984.

Becker, D.L, Carter, M.L. Equipment Available for Sample Screening and On-Site Measurements, Appendices TDD# HQ-8311-04, EPA Contract 68-01-6699, NUS Corporation, May 30, 1984 (includes EPA report and company literature).

METHOD FM-D2: USE OF BONDED SORBENTS FOR PESTICIDE ANALYSIS (METHOD UNDER DEVELOPMENT)

SUMMARY: Method is faster than traditional liquid-liquid extraction. Determines pesticide concentration in water to I ug/l. Requires field laboratory and trained chemist.

METHOD DESCRIPTION: Uses bonded phase extraction column with GC and ECD to determine pesticide concentrations in water. The procedure involves passing 100 ml of filtered ground water through a cyclohexyl-bonded phase extraction column with 1 ml ethylacetate as the elution solvent. The extract injected into GC for analysis. Identification and quantitation are performed by comparing peak heights and areas to standards.

APPLICATION: Determination of pesticide concentrations in water.

LIMITATIONS: Possible interferences from other electron capturing species.

INSTRUMENTATION USED: Varian Model 3700 GC equipped with a nickel electron capture detector and a glass capillary column.

PERFORMANCE SPECIFICATION

DETECTION LIMITS: 1 ug/l for most pesticides

SELECTIVITY: Separate peaks, compounds usually easily identified. Occasional interferences from other electron capturing species.

ACCURACY: Spike recoveries for 5 replicates of 9 compounds = 79%-105%

PRECISION/REPEATABILITY: Coefficient of variation for 5 replicates = 4%

COMMENTS: Performance data are available for recovery as a function of solvent, column, flow rate and type of water.

USE

LOCATION USE: Not Available

EPA SITE NUMBER (CERCLIS): Not Applicable

MATRIX: Water

PREPARATION, MAINTENANCE AND CLEANUP: Columns are disposable. Columns are conditioned prior to use with methanol followed by water or buffer. After the sample has been added, interferences are washed off the column with distilled water, and the sample eluted by a solvent.

ANALYSIS TIME: Sample preparation = 10-15 min; analysis = 20-30 minutes.
CAPITAL COST: $4,000.00 - $20,000.00 for GC

CALIBRATION: Standards were made of high purity solvents with various concentrations of seven pesticides. This is used to spike water samples. The spikes were used for identification and quantitation.

COMMENTS: Interpretation of results requires a trained chemist. Columns must not be allowed to dry out prior to adding sample.

PROTOCOL AVAILABLE: No

SOURCE

TECHNICAL CONTACT: Patricia Gardner

AFFILIATION: Analytichem International

TELEPHONE: 1-(800) 421-2825

PREPARED: April 22, 1987

BIBLIOGRAPHY

Andrews, J.S. and Good, T. J., "Trace Enrichment of Pesticides Using Bonded-Phase Sorbents," American Laboratory, April 1982.

METHOD FM-D3: USE OF BONDED SORBENTS FOR SEMI-VOLATILE ANALYSIS (METHOD UNDER DEVELOPMENT)

SUMMARY: Method is faster than traditional liquid-liquid extraction. Determines most PAHs and phenols in water to 20 ug/l. Poor recovery of phenol (<27%). Analysis can be performed in a field laboratory.

METHOD DESCRIPTION: Uses bonded phase extraction column with GC/MS analysis to determine polycyclic aromatic hydrocarbons (PAHs) and phenols concentrations in water. The procedure involves passing 50-100 ml of filtered ground water through a cyclohexyl-bonded phase extraction column with a solvent, methanol or acetonitrile and methylene chloride, and injecting an aliquot directly into GC for analysis. Response factors were developed using standard mixtures and used to convert mass spectra to quantities based on area of internal standard base peak.

APPLICATION: Determination of concentrations of many phenols and PAHs plus benzothrophene, dibenzofuran, and some nitrogen heterocycles.

LIMITATIONS: Possible interferences from other electron capturing species. Does not detect some phenols and nitrogen heterocycles (e.g., phenol and 2,4-dimethylpyridine).

INSTRUMENTATION USED: Finnigan OWA 1020 computerized capillary gas chromatograph/quadrupole mass spectrometry system (GC/MS) with 30-m fused silica column.

PERFORMANCE SPECIFICATION

DETECTION LIMITS: Less than 20 ug/l for most phenols and PAHs.

SELECTIVITY: Separate peaks, easily identifiable

ACCURACY: Spike recovery for phenolic compounds (except phenol) = 80%-105%. Spike recovery for PAHs and other neutral compounds = 51%-111%, for nitrogen heterocycles = 3%-197%, for phenol=0%-27%.

PRECISION/REPEATABILITY: Standard Deviation (SD) of five replicates: Phenols (except phenol) SD = 5.2%-25.6%. PAHs and neutral compounds; SD = 2.3%-15.8%. Nitrogen heterocycles; SD = 4.4%-32.2%.

COMMENTS: Performance data given for method using acetonitrile and methyl chloride solvent.

USE

LOCATION USED: Not Available

EPA SITE NUMBER (CERCLIS): Not Applicable

MATRIX: Water

PREPARATION, MAINTENANCE AND CLEANUP: Columns are not reusable and must be discarded.

ANALYSIS TIME: Sample preparation = 10-15 min, analysis = 20-30 min/sample

CAPITAL COST: $85,000 - $100,000 for GC/MS; $1 - extraction column

CALIBRATION: Standards are made of high purity solvents with 0.0001 ug/l of various contaminants added. These standards are used to spike water samples to obtain various concentrations. The spikes are used for identification and quantitation.

COMMENTS: Interpretation of results requires a trained chemist. Capital cost varies depending on the GC/MS purchased. HPLC analysis is alternative to GC/MS.

PROTOCOL AVAILABLE: No

SOURCE

TECHNICAL CONTACT:	Patricia Gardner
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TELEPHONE:	1-(800) 421-2825
PREPARED:	April 21, 1987

BIBLIOGRAPHY

Rostad, C.E., Pereiru, W.E., and Rutcliff, S.M., "Bonded-Phase Extraction Column Isolation of Organic Compounds in Ground Water at a Hazardous Waste Site," Analytical Chemistry, Vol. 58, No. 14, p. 2856-2860. December 1984.

Chladek, E and Marano, R.S. "Use of Bonded Phase Silica Sorbents for the Sampling of Priority Pollutants in Wastewaters," Journal of Chromatographic Science, Vol. 22, p. 313-320. August 1984.

METHOD FM-D4: IMMUNOASSAYS FOR TRACE ORGANIC ANALYSIS (METHOD UNDER DEVELOPMENT)

SUMMARY: Alternative (inexpensive and simple) analytical technique incorporating monoclonal antibodies for the quantitative measurement of organic chemicals in ground water & other media. Method for individual chemicals in varying stages of development; development nearly complete for pentachlorophenol. Preliminary data indicates sensitivities equal to GS or HPLC methods.

METHOD DESCRIPTION: Developmental stage technique which is a competitive inhibition enzyme immunoassay (EIA) that incorporates monoclonal antibodies produced via cell fusion (hybridoma) technology. The EIA steps include the addition of reagents (e.g., antibodies) and samples/standards to a microtiter plate, a 3-hour incubation, and spectrophotometric analysis of microtiter plate. A colorimetric reaction indicates the concentration of target compound by reference to a standard curve.

APPLICATION: Inexpensive, rapid and high volume anlysis of organic compounds in aqueous media. Solid phase or oily samples must be extracted into polar solvents prior to introduction into the assay. Antibodies can distinguish stereoisomers.

LIMITATIONS: Limited field studies to date. Antibodies methods cannot detect organic compounds of molecular weight less than 100 atomic units.

INSTRUMENTATION USED: Automated spectrophotometer (microtiter plate reader) IBM compatible personal computer, printer and software package.

PERFORMANCE SPECIFICATION

DETECTION LIMIT: 25 ug/l for pentachlorophenol without sample concentration; depends upon binding strength of antibody for target compound and is compound specific.

SELECTIVITY: Each assay is highly selective for a single compound with minor or no cross-reactivity. Antiboides can also be developed which exhibit broad cross-reactivity with compounds of similar structure.

ACCURACY: Not available

PRECISION/REPEATABILITY: Not available; preliminary results show + 10-20% variability between samples.

COMMENTS: Each sample must be tested in triplicate to provide statistically valid results. Performance data is being compiled in conjunction with US EPA-EMSL Las Vegas.

USE

LOCATION USED: Mobile laboratory and laboratory setting.

EPA SITE NUMBER (CERCLIS): N/A

MATRIX: Water, soil

PREPARATION, MAINTENANCE AND CLEANUP: Antibodies recognize and bind to specific chemical structures, so sample cleanup & purification may not be critical in many cases. Sample preparation for soil samples requires extraction (or solvent exchange) into a polar solvent prior to introduction into the immunoassay. Aqueous samples can usually be analyzed directly or concentrated using reverse phase adsorbtion columns.

ANALYSIS TIME: 4 hours per plate (up to 24 samples per plate); several plates may be run concurrently.

CAPITAL COSTS: $22,000 for complete system, including spectrophotometer, computer (can purchase without), software, printer, plate washer, micropipetters.

CALIBRATION: Prepare a standard curve & controls along with samples. The standard curve is developed using known concentrations of the target compound & occupies 18 wells on the plate.

COMMENTS: Immunoassays are simple to perform; Highly trained operators not needed. Methods under development for dioxin, (2,3,7,8-TCDD) pesticides, PCBs, benzene, phenol,etc. Studies to compare method to other methods will be performed.

PROTOCOL AVAILABLE: Yes

SOURCE

TECHNICAL CONTACT: Joseph Paladino, Product Manager
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TELEPHONE: (412) 722-5602
PREPARED: May 19, 1987
BIBLIOGRAPHY: Technical Product Description (Corporate Literature), personal conversation

METHOD FM-D5: USE OF FIBER OPTIC SENSORS IN ENVIRONMENTAL MONITORING (METHOD UNDER DEVELOPMENT)

SUMMARY: *In situ* monitoring technique for various contaminants to ug/l concentrations in ground water, air, & soil. Few fully developed applications, but appears to offer significant advantages over conventional sampling techniques.

METHOD DESCRIPTION: Optical fibers are used either as a "light tube" in remote fiber spectroscopy mode (RFS) or as a "sensor" in fiber optical chemical sensing mode. RFS provides light transmission over large distances with low light loss; transmission depends on the core having a higher index of refraction than the cladding. Configurations include use of laser-induced fluorescence, absorption spectroscopy, raman spectroscopy, & multivariate spectral analysis. A fiber optical chemical sensor (FOCS) unit includes a chemical-specific sensor & can operate in at least three modes: fluorescence, absorption & refraction. A FOCS can respond to a class of compounds or a single species.

APPLICATION: *In situ*, real-time monitoring of environmental contamination. Particularly suited for inaccessible or hazardous situations (e.g., ground water, spill cleanup monitoring).

LIMITATIONS: A different FOCS is needed for each pollutant to be monitored, increasing development costs (but many can be bundled in a < 1/2" package). Both techniques are still developmental.

INSTRUMENTATION USED: Optical fibers with special coatings attached to various analytical instruments, e.g., a portable laser spectrometer, fluroesence spectrometer, etc.

PERFORMANCE SPECIFICATION

DETECTION LIMIT: RFS: Low ug/l range for aromatic gasoline components using laser-induced fluorescence; ug/l for organophosphorus pesticides & other compounds using raman spectroscopy.
FOCS: low ug/l range for chloroform.

SELECTIVITY: RFS: fluorescence spectra showed good separation of contaminants from background.
FOCS: sensors for specific chemicals used, thus selectivity is high.

ACCURACY: Improved over conventional sampling techniques (i.e., loss of analyte during well pumping and sample storage in conventional techniques).

PRECISION/REPEATABILITY: Results of repeated analyses for chloroform from one well all fall within 3% using an organic chloride FOCS.

COMMENTS: Organic chloride FOCS developed by EPA are integrating devices. Slope of the response curve can be directly related to sample concentrations. Chemical specificity of FOCS allows simplification of the spectrometer. Independent GC/MS analysis can be used to confirm results.

USE

LOCATION USED: Henderson, NV

EPA SITE NUMBER (CERCLIS): Non-CERCLA

MATRIX: Water, air, soil

PREPARATION, MAINTENANCE AND CLEANUP: Reuse of FOCS requires cleanup (e.g., for organic chloride FOCS, bleach the fluorescent reaction product by raising the source power).

ANALYSIS TIME: NA

CAPITAL COSTS: RFS: Low developmental cost but high operational and equipment cost. FOCS: Individual FOCS elements should cost less than $25. Instruments would cost from $500 to $10,000 depending on configuration (e.g., spectrometer or refractometer, single or multi-channel, etc.).

CALIBRATION: FOCS: Experiments are run with each type of FOCS to produce a calibration curve using precalibrated solutions.

COMMENTS: Limited field experience with most FRS amd FOCS applications. Research & commercialization effort underway by several agencies and companies.

PROTOCOL AVAILABLE: No

SOURCE

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PREPARED:	May 18, 1987

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APPENDIX B

HISTORICAL PRECISION AND ACCURACY DATA CLASSIFIED BY MEDIA BY ANALYTICAL LEVEL

HISTORICAL PRECISION AND ACCURACY TABLES

Introduction

Table B-1	Water:Level III (Other than CLP RAS)
Table B-2	Water:Level III (SW-846)
Table B-3	Water:Level IV
Table B-4	Soil:Level I
Table B-5	Soil:Level II
Table B-6	Soil:Level III
Table B-7	Soil:Level IV
Table B-8	Air: Level I
Table B-9	Air: Level II
Table B-10	Air: Level III
Table B-11	Other Media: Level III

INTRODUCTION

The data in this Appendix have been compiled to assist the reader in selecting an analytical method appropriate for each data use. The methods are classified by media and by analytical levels defined as follows:

- Level I - field screening or analysis using portable instruments. Results are often not compound specific and not quantitative but results are available in real-time.
- Level II - field analysis using more sophisticated portable analytical instruments; in some cases, the instruments may be set up in a mobile or onsite laboratory. There is a wide range in the quality of data that can be generated. Quality depends on the use of suitable calibration standards, reference materials, and sample preparation equipment; and the training of the operator. Results are available in real-time or several hours.
- Level III - all analyses performed in an offsite analytical laboratory using standard, documented procedures. The laboratory may or may not be a CLP laboratory.
- Level IV - CLP routine analytical services (RAS). All analyses are performed in an offsite CLP analytical laboratory following CLP protocols.

Precision and accuracy data are presented in tabular fashion. Footnotes to each table cite the sources of the data and the concentration or concentration range at which the precision and accuracy were determined. When no concentration is cited no concentration information was available in the source material.

Precision is a measure of the variability in repeated measurements of the same sample compared to the average value. Precision is reported as % Relative Standard Deviation (RSD). The lower the % RSD, the more precise the data.

TABLE B-1. HISTORICAL PRECISION AND ACCURACY DATA/WATER[a]

LEVEL III ANALYTICAL TECHNIQUES - METHODS OTHER THAN CLP RAS METHODS

ANALYTES	METHOD (TECHNIQUE)	CONCENTRATION RANGE	PRECISION % RSD	ACCURACY % BIAS
Benzene	624 (GC/MS)	11 ug/l 480 ug/l	16 21	0 -16
	8240 (GC/MS)	5-100 ug/l	21	12
Bromodichloromethane	624 (GC/MS)	8 ug/l 480 ug/l	28 18	-8.8 -6.7
	501.1 (PURGE & TRAP GC/MS)	0.9 ug/l 550 ug/l	66 34	0 -3.8
	501.2 (EXTRACTION GC/MS)	1.8 ug/l 170 ug/l	61 23	33 -19
Bromoform	624 (GC/MS)	9 ug/l 400 ug/l	32 30	-23 10
	501.1 (PURGE & TRAP GC/MS)	4.8 ug/l 550 ug/l	44 41	-27 7.5
	501.2 (EXTRACTION GC/MS)	6 ug/l 170 ug/l	14 15	-23 1.8

TABLE B-1. HISTORICAL PRECISION AND ACCURACY DATA/WATER[a]
(continued)

LEVEL III ANALYTICAL TECHNIQUES - METHODS OTHER THAN CLP RAS METHODS

ANALYTES	METHOD (TECHNIQUE)	CONCENTRATION RANGE	PRECISION % RSD	ACCURACY % BIAS
Chloroform	624	4.5 ug/l	31	2.2
	(GC/MS)	300 ug/l	14	-0.6
	501.1	0.9 ug/l	64	44
	(PURGE & TRAP GC/MS)	550 ug/l	14	-0.02
	501.2	1.8 ug/l	68	-39
	(EXTRACTION GC/MS)	170 ug/l	26	-1.2
Dibromochloromethane	624	8.1 ug/l	13	-3.1
	(GC/MS)	360 ug/l	19	10
	501.1	0.8 ug/l	35	-12.5
	(PURGE & TRAP GC/MS)	550 ug/l	36	4.7
	501.2	1.8 ug/l	37	0
	(EXTRACTION GC/MS)	170 ug/l	13	0.02
Dioxin	613	21 ng/l	25	N.A.
	(GC/MS)	202 ng/l	21	N.A.

TABLE B-1. HISTORICAL PRECISION AND ACCURACY DATA/WATER[a]
(continued)

LEVEL III ANALYTICAL TECHNIQUES - METHODS OTHER THAN CLP RAS METHODS

ANALYTES	METHOD (TECHNIQUE)	CONCENTRATION RANGE	PRECISION % RSD	ACCURACY % BIAS
Methylene Chloride	624 (GC/MS)	7.2 ug/l 480 ug/l	78 52	-17 -25
Toluene	624 (GC/MS)	13.5 ug/l 600 ug/l	19 31	15 -14
	8240 (GC/MS)	25 ug/l 75 ug/l	19	-10
Trichloroethene	624 (GC/MS)	5.4 ug/l 360 ug/l	48 39	44 -2.3
	8240 (GC/MS)	25 ug/l 75 ug/l	24	5
Lead	200.7 (ICP)	42 ug/l 47.7 ug/l	34 5	31 4.4
	239.1 (FLAME AA)	12 ug/l 105 ug/l	5.9 6.7	17 -1.9
	239.2 (FURNACE AA)	10 ug/l 234 ug/l	53 19	-22 -3.1

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[a] Source: Draft Compendium of Information and Performance Data on Routinely Used Measurement Methods (RUMM) - Pilot Phase, RTI/3087/03, prepared for EPA Quality Assurance Management Staff, January 1986. This document should be consulted for more information on individual analytes.

TABLE B-2. HISTORICAL PRECISION AND ACCURACY DATA/WATER

LEVEL III SW-846 METHODS

Method Number	Method Name	Data Source	Range of Recovery (%)	Precision (%)	MDL (mg/l)
ORGANICS:					
8010	Halogenated Volatile Organics	SW 846	75.1 - 106.1	2.0 - 25.1	0.03 - 0.
8020	Aromatic Volatile Organics	SW 846	77.0 - 120	9.4 - 27.7	0.2 - 0.4
8030	Acrolein, Acrylonitrile, Acetonitrile	SW 846	96 - 107	5.6 - 11.6	0.5 - 0.6
8040	Phenols	SW 846	41 - 86	7.9 - 16.5	058 - 2.2
8060	Esters	EPA 606	82 - 94	1.3 - 6.5	0.29 - 3.
8080	Organochlorine Pesticides and PCBs	SW 846	86 - 97	1.3 - 6.5	0.29 - 3.
8090	Nitroaromatics and Cyclic Ketones	SW 846	63 - 71	3.1 - 5.9	0.06/ND
8100	Polynuclear Aromatic Hydrocarbons		NA[b]	NA	NA
8120	Chlorinated Hydrocarbons	SW 846	76 - 99	10 - 25	0.03 - 1.
8140	Organophosphorous Pesticides	SW 846	56.5 - 120.7	5.3 - 19.9	0.1 - 5.0
8150	Chlorinated Herbicides	SW 846	NA	NA	0.1 - 200
8240	Volatile Organics	SW 846	95 - 107	9 - 28	1.6 - 6.9
8250	GC/MS Semivolatiles (Packed Column)		41 - 143	20 - 145	0.9 - 44
8040	GC/MS Semivolatiles (Capillary)		NA	NA	NA
8310	Polynuclear Aromatic Hydrocarbons (HPLC) (Capillary)	SW 846	78 - 116	7.3 - 12.9	0.03 - 2.

TABLE B-2. HISTORICAL PRECISION AND ACCURACY DATA/WATER
(continued)

LEVEL III SW-846 METHODS

Method Number	Method Name	Data Source	Range of Recovery (%)	Precision (%)	MDL (mg/l)
INORGANICS:	Metals (ICAP)	EPA 200.7	NA	3 - 21.9 (RSD)	1.3 - 75 Mg/l
	Metals (FLAME) 7000 Series	EPA 200	NA	NA	0.01 - 5
7000 Series	Metals (FLAMELESS/GF)	EPA 200	NA	NA	0.001 - 0.2 Mg/l
7470	Metals (MERCURY)	EPA 245.2	87 - 125	0.9 - 4.0	0.0002
9010	Cyanides	EPA 335.2	85 - 102	0.2 - 15.2	0.02 Mg/l
9030	Sulfides	EPA 376.1	NA	NA	1 Mg/l

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a. For water only

b. NA = Not Available

NOTES: Method Detection Limit (MDL) as listed on this table is the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero.

Accuracy, presented as an average percent recovery, was determined from replicate (10-25) analyses of water and wastewater samples fortified with known concentrations of the analyte of interest or near the detection limit. In most cases this was less than 10 times the MDL.

Precision data are used to measure the variability of these repetitive analyses reported as a single standard deviation or, as a percentage of the recovery measurements. For presentation purposes accuracy, precision and MDL information is presented as an average range of individual values for every analyte covered by the procedure. If specific information on a particular compound is required, the specific analytical method cited should be consulted.

TABLE B-3. HISTORICAL PRECISION AND ACCURACY DATA/WATER[a]

LEVEL IV ANALYTICAL TECHNIQUES - CLP RAS METHODS

ANALYTES	TECHNIQUE	CONCENTRATION RANGE	PRECISION % RSD	ACCURACY % Bias
Volatiles[b]	Purge & Trap GC/MS	N.A.[c]		
Methylene chloride			56	+36.6
1,1-Dichloroethene			20	-26.3
1,1-Dichloroethane			13	-46.4
Trans-1,2-Dichloroethene			31	-21.7
Chloroform			12	-21.1
1,2-Dichloroethane			13	+2.4
1,1,1-Trichloroethane			19	-41.0
Carbon Tetrachloride			12	-32.1
1,1,2,2-Tetrachloroethane			11	-5.8
Bromodichloromethane			19	-13.0
1,2-Dichloropropane			18	-12.9
Trans-1,3-Dichloropropene			31	-41.2
Trichloroethene			17	-22.8
Dibromochloromethane			14	-3.3
1,1,2-Trichloroethane			11	-7.0
Benzene			12	-3.3
Cis-1,3-Dichloropropene			22	-35.5
Bromoform			16	+6.5
Tetrachloroethene			13	-42.5
Toluene			14	-23.3
Chlorobenzene			14	-15.9
Ethyl Benzene			4	-31.9
Semivolatiles[d]	GC/MS	N.A.[c]		
bis(2-Chloroethyl)ether			24	-16
2-Chlorophenol			29	-21
1,3-Dichlorobenzene			24	-48
1,4-Dichlorobenzene			21	-25
1,2-Dichlorobenzene			29	-28
2-Methylphenol			29	-30
bis(2-Chloroisopropyl)ether			25	-22

TABLE B-3. HISTORICAL PRECISION AND ACCURACY DATA/WATER[a]
(continued)

LEVEL IV ANALYTICAL TECHNIQUES - CLP RAS METHODS

ANALYTES	TECHNIQUE	CONCENTRATION RANGE	PRECISION % RSD	ACCURACY % Bias
Semivolatiles[d]	GC/MS	N.A.[c]		
4-Methylphenol			33	-36
N-Nitroso-di-n-propylamine			31	+0.3
Nitrobenzene			32	-23
Isophorone			23	-8
2-Nitrophenol			30	-21
bis(2-Chloroethoxy)methane			34	-2.6
2,4-Dichlorophenol			29	-20
1,2,4-Trichlorobenzene			30	-47
Naphthalene			44	-38
4-Chloro-3-methylphenol			26	-32
2,4,6-Trichlorophenol			25	-17
2-Chloronapthalene			24	+3.4
Acenapthene			28	-12
2,4-Dinitrophenol			24	-23
2,4-Dinitrotoluene			34	-33
2,6-Dinitrotoluene			25	-48
4-Chlorophenyl-phenylether			34	+12
Fluorene			25	-24
4,6-Dinitro-2-methylphenol			30	-13
4-Bromophenyl-phenylether			32	-0.1
Hexachlorobenzene			36	-42
Pentachlorophenol			31	-24
Phenanthrene			21	-28
Fluoranthene			42	-15
Benzo(b)fluoranthene			39	-10
Benzo(a)pyrene			42	-29

TABLE B-3. HISTORICAL PRECISION AND ACCURACY DATA/WATER[a]
(continued)

LEVEL IV ANALYTICAL TECHNIQUES - CLP RAS METHODS

ANALYTES	TECHNIQUE	CONCENTRATION RANGE	PRECISION % RSD	ACCURACY % Bias
Metals[a]				
Aluminum	ICP	1000-3000 ug/l	9.1	-4.3
Antimony	ICP	180-600 ug/l	11	-9.2
Arsenic	Furnace AA	50-150	9.4	-8.3
Barium	ICP	800-1500	6.8	-3.9
Beryllium	ICP	30-45	15	+3.7
Cadmium	ICP	25-50	12	-3.3
Calcium	ICP	1000-30000	6.0	-1.6
Chromium	ICP	50-150	9.8	-2.6
Cobalt	ICP	200-1000	6.7	-2.9
Copper	ICP	125-250	6.7	-1.1
Iron	ICP	200-800	10.4	+6.5
Lead	Furnace AA	30	32	-0.7
Magnesium	ICP	10000-40000	6.6	-2.5
Maganese	ICP	30-150	6.2	-1.0
Mercury	Cold Vapor	5-20	18.8	-14.4
Nickel	ICP	160	9.0	-2.5
Potassium	ICP	10000-20000	16.2	-12.1
Selenium	Furnace AA	50	8.7	-5.7
Sodium	ICP	10000-45000	8.7	-2.8
Thallium	Furnace AA	80-100	17.7	-4.2
Tin	ICP	160	N.A.[c]	-2.5
Vanadium	ICP	60-200	7.6	-0.46
Zinc	ICP	50-800	9.1	+3.0

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[a] Source: Quality Control in Remedial Site Investigation: Hazardous and Industrial Solid Waste Testing, Fifth Volume, ASTM STP 925, C.L. Perket, Ed., American Society for Testing Materials, Philadelphia, 1986.

b. Volatile precision and accuracy data from 26-34 laboratories' results on quarterly blind performance evaluation samples; 29-152 data points for each compound.

c. N.A. = Not Available.

d. Semivolatile precision and accuracy data from 1985 preaward program data; 22-227 data points for each compound.

e. Metals precision and accuracy data is based on performance evaluation sample results from 18 laboratories; number of data points is not given.

TABLE B-4. HISTORICAL PRECISION AND ACCURACY DATA/SOILS

LEVEL I FIELD SCREENING TECHNIQUES

MEASUREMENT	INSTRUMENT (TECHNIQUE)	INSTRUMENT RANGE	INSTRUMENT PRECISION [b]	INSTRUMENT ACCURACY [c]
Resistivity	Bison 2390 T/R (Resistivity meter)	0-1999 millivolts	at 1% range setting, 0-5% of full scale	2% of measured value
Terrain Conductance	EM 31 (conductivity)	0-1000 millimhos/meter	2% of full scale	5% at 20 millimhos/meter
Terrain Conductance	EM 34-3 (conductivity)	0-300 millimhos/meter	2% of full scale	5% at 20 millimhos/meter
Magnetic Field Intensity	EDA - Omni IV (Magnetometer)	18000-11000 gammas	0.02 gamma	1 gamma at 50000 gammas at 23°C
Subsurface Lithology Changes	SIR-8 (Ground Penetrating Radar)	1-81 dielectric constant	N/A[d]	N/A[d]
Subsurface Lithology Changes	EG+G 1225 (Seismograph)	0-2000 milliseconds	N/A[d]	0.01%

TABLE B-4. HISTORICAL PRECISION AND ACCURACY DATA/SOIL[a]
(continued)

LEVEL I FIELD SCREENING TECHNIQUES

MEASUREMENT	INSTRUMENT (TECHNIQUE)	FIELD SCREENING RESULTS in ppm (X)	CLP RESULTS in ppm (Y)	ACCURACY[e] (% Bias)
Total Volatile Organics	PHOTO VAC (GC/Photoionization)	11.4	26.9	-57.6
		22.0	32.8	-32.9
		56.0	129.7	-56.8
		139	228.0 & 258.0	-42.8
		70.0	126.7	-44.8
		24.9	2823.0	+99.1
		60.0	53.3	+12.6
		6.6	0.056	+116.9
		12.1	0.032	+377.1
		8.7	0.024	+361.5

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a. Source: Manufacturers' manuals unless otherwise cited. Mention of specific models does not constitute an endorsement of these instruments.

b. Precision refers to reproducibility of meter or instrument reading as cited in instrument specifications.

c. Accuracy refers to instrument specifications unless otherwise cited.

d. N.A. = Not available.

e. Accuracy of PhotoVac field screening results calculated by assuming that CLP results on the same samples were completly accurate. % Bias = $100 \frac{(X-Y)}{Y}$. Source of these data is CDM project files.

TABLE B-5. HISTORICAL PRECISION AND ACCURACY DATA/SOIL[a]

LEVEL II FIELD ANALYSIS

LEVEL II FIELD TECHNIQUES

ANALYTES	INSTRUMENT (TECHNIQUE)	FIELD RESULTS IN ppm (x)	CLP RESULTS IN ppm (y)	ACCURACY[b] % BIAS
PCBs	HNu 301 (GC/Electron Capture)	6.0	22.0	-72.7
		6.0	6.1	-1.6
		6.0	510.0	-98.8
		9.0	3.9	+56.7
		13.0	3.0	+333.3
		14.0	3.1	+351.6
		14.0	23.5	-40.5
		21.0	8.1	+159.3
		35.0	7.7	354.5
		41.0	2.1	+1,852
		48.0	11.0	+336.3
		50.0	460.0	-89.1
		65.0	23.1	+181.4
		67.0	18.7	+258.3
		92.0	75.0	22.7
		95.0	30.0	+216.7
		11	12.3	-10.6
		202	99.0	+104.0
		269	370.0	-27.3
		286	80.5	+255.3
		1215	640.0	+90.0
		1647	1040.0	+58.4
		3054	9,300	-67.2

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a. Source: CDM Project files.

b. Source: Accuracy calculated by assuming that CLP results on the same samples were completely accurate.
$\% \text{ Bias} = 100 \frac{(x-y)}{y}$

TABLE B-6. HISTORICAL PRECISION AND ACCURACY DATA/SOIL[a]

LEVEL III ANALYTICAL TECHNIQUES - METHODS OTHER THAN CLP RAS METHODS

ANALYTE	METHOD (TECHNIQUE)	CONCENTRATION RANGE	PRECISION % RSD	ACCURACY % BIAS
Dioxins	8280 (HPLC/LRMS)	5 ppb	6-30	N.A.
		125 ppb	3-10	N.A.
	JAR EXTRACTION GC/MS	1 ppb	20	0
		10 ppb	10	-18

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a. Source: Draft Compendium of Information and Performance Data on Routinely Used Measurement Methods (RUMM) - Pilot Phase, RTI/3087/03, prepared for EPA Quality Assurance Management Staff, January 1986. This document should be consulted for more information on individual analytes.

TABLE B-7. HISTORICAL PRECISION AND ACCURACY DATA/SOILS[a]

LEVEL IV ANALYTICAL TECHNIQUES - CLP RAS METHODS

ANALYTES	TECHNIQUE	CONCENTRATION RANGE	PRECISION % RSD	ACCURACY % Bias
Volatiles[b]	Purge & Trap GC/MS	N.A.[c]		
Chloroform			8.0	-0.1
1,2-Dichloroethane			13.1	+11.1
Dibromochloromethane			35.0	-12.0
Benzene			32.1	-10.3
Bromoform			16.6	-12.1
2-Hexanone			16.6	-45.5
Toluene			13.8	+13.7
Chlorobenzene			21.2	+13.2
Semivolatiles[d]	GC/MS	N.A.[c]		
1,4-Dichlorobenzene			27	-51
Nitrobenzene			21	-48
Isophorone			24	-47
2-Nitrophenol			35	-36
2,4-Dichlorophenol			31	-59
1,2,4-Trichlorobenzene			28	-43
Penta Chlorophenol			17	-48
Pyrene			25	-15
2-Methylnahthalene			26	-42
bis-(2-Ethylhexyl)phthalate			33	-2
Phenol			38	-27
Acenaphthylene			26	-27
Diethyphthalate			16	-20
Dioxin[e]				
2,3,7,8-TCCD		1-10 ug/kg	15	-11.5

TABLE B-7. HISTORICAL PRECISION AND ACCURACY DATA/SOILS[a]
(continued)

LEVEL IV ANALYTICAL TECHNIQUES - CLP RAS METHODS

ANALYTES	TECHNIQUE	CONCENTRATION RANGE (ug/kg)	PRECISION % RSD	ACCURACY % Bias
Metals[b]				
Aluminum	ICP	2-22600	14.4	-78.8
Cadmium	ICP	5.5-20	33.3	+2.9
Calcium	ICP	2664-29000	N.A.[c]	-4.2
Chromium	ICP	8.5-29600	7.8	-6.1
Copper	ICP	33-109	11.2	-2.5
Iron	ICP	5028-113000	10.7	-27.0
Lead	Furnace AA	11.5-714	9.2	-2.2
Magnesium	ICP	2428-7799	7.5	-10.6
Manganese	ICP	73.5-785	9.4	-15.1
Mercury	Cold Vapor	1.1-26.5	25.0	-9.1
Nickel	ICP	44-67	15.0	-17.0
Tin	ICP	N.A.[c]	44.1	N.A.[c]
Zinc	ICP	19-1720	5.8	-6.2

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a. Source: Quality Control in Remedial Site Investigation: Hazardous and Industrial Solid Waste Testing, Fifth Volume, ASTM STP 925, C.L. Perket, Ed., American Society for Testing Materials, Philadelphia, 1986.

b. Volatiles precision and accuracy data is based on 1985 preaward analysis results from laboratories awarded contracts; 6-14 data points for each compound.

c. N.A. = Not Available.

d. Semivolatiles precision and accuracy data is based on 1985 preaward analysis results; 9-20 data points for each compound.

e. Dioxin precision for accuracy data is based on results of four performance evaluation samples including 120 data points.

f. Metals precision and accuracy data is based on performance evaluation sample results from 18 laboratories; number of data points is not given.

TABLE B-8. HISTORICAL PRECISION AND ACCURACY DATA/AIR[a]

LEVEL I FIELD SCREENING TEHCNIQUES[b]

ANALYTES	INSTRUMENT (TECHNIQUE)	INSTRUMENT RANGE	INSTRUMENT SENSITIVITY[c]	INSTRUMENT PRECISION [c]
Organics	Century OVA-128 (Flame Ionization)	0.1 - 1000 ppm Methane	0.1 ppm Methane	N.A.[d]
Organics	HNu PI-101 (Photoionization)	0.1 - 2000 ppm Benzene	0.1 ppm Benzene	1% of full scale deflection
Organics	AID - 710 (Flame Ionization)	0.1 - 2000 ppm Methane	0.1 ppm Methane	N.A.[d]
Organics	PhotoVac (GC-Photoionization)	N.A.	0.001 ppm Benzene	N.A.[d]

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a. Source: Manufacturers' manuals unless otherwise cited. Mention of specific models does not constitute an endorsement of these instruments.

b. It is difficult to differentiate between Level I and Level II techniques and instrumentation. Several instruments may be used at both levels.

c. Sensitivity and precision refer to instrument specifications.

d. N.A. = Not Available.

TABLE B-9. HISTORICAL PRECISION AND ACCURACY DATA/AIR[a]

LEVEL II FIELD TECHNIQUES[b]

ANALYTES	INSTRUMENT (TECHNIQUE)	INSTRUMENT RANGE	INSTRUMENT SENSITIVITY[c]	INSTRUMENT PRECISION[c]
Organics Compound-Specific	Miran IB (Infrared)	Compound Dependent 0-2000 ppm	N.A.[d]	N.A.[d]
Organics, Compound-Specific	Century OVA-128 (GC/Flame Ionization)	1-1000 ppm Methane	N.A.	N.A.
Organics, Compound-Specific	PhotoVac (GC-Photo-ionization)	N.A.	0.001 ppm Benzene	N.A.
Organics, Compound-Specific	SCENTOR (Argon Ionization or Electron Capture)	N.A.	0.001 ppm Benzene	N.A.
Mercury	Gold film Mercury Analyzer	N.A.	less than 0.01 ppm	N.A.

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a. Source: Manufacturers' manuals. Mention of specific models does not constitute an endorsement of these instruments.

b. It is difficult to differentiate between Level I and Level II techniques and instrumentation. Several instruments may be used at both levels.

c. Sensitivity and precision refer to instrument specifications.

d. N.A. = Not Available.

TABLE B-10. HISTORICAL PRECISION AND ACCURACY DATA/AIR[a]

LEVEL III ANALYTICAL TECHNIQUES - METHODS OTHER THAN CLP RAS METHODS

ANALYTES	METHOD (TECHNIQUE)	CONCENTRATION RANGE	PRECISION % RSD	ACCURACY % BIAS
Benzene	CRYOGENIC TRAP/GC	3.9 ppb	4.0	N.A.
		93 ppb	5.1	N.A.
	TENAX GC/MS	7.8 ug/m3	11	N.A.
		4.5 ug/m3	21	N.A.
Toluene		10.8 ppm	5.11	N.A.
Trichloroethene		3.5 ppb	4.1	N.A.
		84 ppb	3.7	N.A.
Vinyl Chloride		7.8 ppb	6.37	N.A.
Lead	40 CFR 50, APP G	0.6 ug/m3	8.6	0
	(FLAME AA)	8.01 ug/m3	3.9	-3.6

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a. Source: Draft Compendium of Information and Performance Data on Rountinely Used Measurement Methods (RUMM) - Pilot Phase, RTI/3087/03, prepared for EPA Quality Assurance Management Staff, January 1986. This document should be consulted for more information on individual analytes.

TABLE B-11. HISTORICAL PRECISION AND ACCURACY DATA/OTHER MEDIA[a]

LEVEL III ANALYTICAL TECHNIQUES - METHODS OTHER THAN CLP RAS METHODS

ANALYTE	METHOD (TECHNIQUE)	MEDIUM	CONCENTRATION RANGE	PRECISION % RSD	ACCURACY % BIAS
Lead	6010 (ICP)	Oil Waste	1.0 mg/kg -2.5 mg/kg	3.1 22	-10 -20
		Solid Waste	50 mg/kg 75 mg/kg	10 3.7	3.4 -0.8
	Solid	Sludge	5 mg/kg 20 mg/kg	2 11	0 55

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a. Source: Draft Compendium of Information and Performance Data on Routinely Used Measurement Methods (RUMM) - Pilot Phase, RTI/3087/03 prepared for EPA Quality Assurance Management Staff, January 1986. This document should be consulted for more information on individual analytes.

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APPENDIX C

USER COMMENT FORM

FIELD SCREENING METHODS CATALOG (FSMC)

USER COMMENT FORM

Your comments and suggestions are important to us. Comments may relate to the software, user manual, or mini-guide, or may be a request for an addition or correction to the method descriptions, and additional copies of the system discs. Please return this form to:

FSMC System Coordinator
Office of Emergency and Remedial Response
Analytical Operations Branch (WH-548-A)
U.S. Environmental Protection Agency
Washington, DC 20460

Please provide the following information so that we may follow up if necessary.

Your name: ______________________________

Address: ______________________________

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Phone number: ______________________________

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